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THE PRIMARY VOLATILE PRODUCTS OF THE CARBONIZATION OF COAL

[A Sequel to Bulletin 1, The Volatile Matter of Coal]

BY

GUY B. TAYLOR

AND

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WASHINGTON

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THE PRIMARY VOLATILE PRODUCTS OF THE CARBONIZATION OF COAL.

By GUY B. TAYLOR and HORACE C. PORTER.

INTRODUCTION.

This report is a continuation of the work described in Bureau of Mines Bulletin 1 on the character of the volatile matter of coal. The experimental methods have been refined and more precise results obtained.

The importance of an exact knowledge of the reactions taking place when coal is heated need not be dwelt upon at length. The subject of carbonization is important in all industrial applications of coal, whether it be in gas manufacture or coke making, where it is paramount, or in those processes where coal is burned, such as in boiler furnaces, gas producers, and metallurgical furnaces. The results presented here are more or less of a fundamental character—a study of basic phenomena in carbonization, rather than of the ultimate results of these phenomena as a whole.

NATURE OF THE PROBLEM.

When coal is heated with exclusion of oxygen, chemical reactions occur that are highly complex and a variety of new substances appears in place of the original coal. These new substances are in three phases—the solid altered residue, or coke; liquid products, or water and tars; and gas. The yield and character of these (coke, tar, water, and gas) from any coal may be varied widely by changing the external conditions of temperature, pressure, time, and contact surfaces. The importance of these factors on the composition of the ultimate products of coal carbonization needs to be emphasized. Lewes [a] makes the following just criticism:

> We make elaborate tables of the composition of gases and tars at various distillation temperatures, but the only information that they give us is what is left undecomposed under unknown and varying conditions, the only certain factors being that the heat was nowhere above that which we are pleased to call the temperature of distillation.

In carbonization in bulk it is doubtful whether any of the final products are liberated as such from the coal. Secondary reactions among the primary products occur easily and follow well-known physical-chemical laws. But the components of such a system are so many

[a] Lewes, V. B., The carbonization of coal, 1913, p. 130.

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and complex that application of the laws of chemical statics and dynamics to carbonization processes as a whole is extremely difficult. The process may, however, be divided into its various steps, as follows: First, liberation of primary volatile matter at low temperatures, with alteration of the coal substance into a partly carbonized solid; second, liberation of volatile products from the altered residue by subjecting it to higher temperatures; and finally, thermal reactions among the primary volatile products. The last is by far the most important in its bearing upon ultimate by-products from the coal.

It has been the object of this investigation to study the primary volatile products liberated from coal by heat, avoiding secondary reactions as far as practicable in obtaining these primary products, and the apparatus and methods used have been planned with this end in view. Secondary reactions have been largely prevented by reducing to a minimum the time of contact of the volatile matter with heated surfaces.

The results of the investigation include information on the amount and character of the primary volatile products from four types of American coals; data showing the effects of temperature and time of heating on the character of the solid residue and volatilized products from coal; and experimental evidence of the importance and general effect of secondary reactions of the primary volatile matter on the final products of carbonization.

TYPES OF COAL USED IN EXPERIMENTS.

The four kinds of coal used were representative of distinct types or classes of coal. All of the samples had been kept since mining in closed containers, as ¼-inch to ½-inch lumps. Their composition was as follows:

Results of analyses of coals used in experiments.

AIR-DRIED (AS USED).

	Pittsburgh (No. 650).	New River, W. Va. (No. 674).	Illinois (No. 48).	Wyoming (No. 43).
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
Moisture	0.93	0.60	2.07	5.98
Volatile matter	33.02	21.60	35.21	41.47
Fixed carbon	56.98	74.57	53.55	46.09
Ash	9.07	3.23	9.17	6.46
Sulphur	1.81	.68	1.17	1.02
Carbon	76.73	85.92	71.80	65.63
Hydrogen	5.00	4.76	4.79	5.27
Oxygen	5.86	3.96	11.52	20.13
Nitrogen	1.53	1.45	1.55	1.49
Calorific value (calories)	7,601	8,412	6,980	6,410
CALCULATED TO DRY, ASH-FREE MATERIAL.				
Volatile matter	36.69	22.42	39.70	47.35
Carbon	85.25	89.30	80.91	74.94
Hydrogen	5.45	4.88	5.14	5.26
Oxygen	5.59	3.60	10.89	16.93

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DESCRIPTION OF SAMPLES.

New River, W. Va. (*lab. No. 674*).—Mine sample from the Sun mine, Sewell bed, Fayette County, W. Va. (New River district), kept in closed container 4 years after mining.

Pittsburgh (*lab. No. 650*).—Car sample from the Keystone Shaft mine, Madison, Westmoreland County, Pa. (Pittsburgh bed), kept in closed container 1 year after mining.

Illinois (*lab. No. 48*).—Mine sample from the Dering No. 11 mine, West Frankfort, Franklin County, Ill., kept in closed container 5 years after mining.

Wyoming (*lab. No. 43*).—Mine sample from Dietz No. 2 mine, Sheridan County, Wyo., bed No. 2, kept in closed container 5 years after mining.

METHODS USED IN EXPERIMENTS.

TEMPERATURE MEASUREMENTS.

Calibrated thermoelements of copper and constantan, and of platinum and platinum-rhodium (10 per cent rhodium), were used with direct-reading Siemens and Halske millivoltmeters. Readings could be made to within 2° C.

ANALYSIS OF GASES.

The gases were collected and measured over mercury. Volumes of gases were measured at room conditions—that is, at a temperature of 25° C. and a pressure of 740 millimeters, the gases being saturated with water vapor.

On account of the complex nature of the gases a special form of apparatus was necessary. After an exhaustive trial with all the methods known for estimating hydrogen that depend on the catalytic action of palladium, it was found that for the gases evolved at lower temperatures these methods were unsatisfactory. The Jaeger [a] method of fractional combustion with copper oxide worked very well.

In the gas-analysis apparatus (fig. 1) the Bone and Wheeler [b] method of absorption has been combined with the usual form of compensated burette, *A*, a slow-combustion pipette, *B*, and a copper-oxide U tube, *C*. With this apparatus, the construction and manipulation of which has been fully described by Taylor,[c] a complete analysis, including the estimation of hydrogen, may be made with the same sample.

Absorptions were made with a few c. c. of each reagent introduced under the mercury in *D* or *E*. As a rule the sample upon introduction into the burette blackened the mercury, which made the reading of

[a] See Dennis, L. M., Gas analysis, 1913, p. 201.

[b] Bone, W. A., and Wheeler, R. V., An accurate form of gas-analysis apparatus for commercial and other purposes: Jour. Soc. Chem. Ind., vol. 27, 1908, pp. 10–11.

[c] Taylor, G. B., An apparatus for the analysis of complex gas mixtures: Jour. Ind. and Eng. Chem., vol. 6, 1914, pp. 845–848.

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the initial volume difficult, but after treatment with acidified copper sulphate solution to remove sulphides, further readings were not interfered with. The successive absorbing agents were: For H_2S and NH_3, acid $CuSO_4$ solution; for CO_2, 30 per cent KOH solution; for unsaturated hydrocarbons (and benzol), fuming sulphuric acid; for O_2, alkaline pyrogallol.

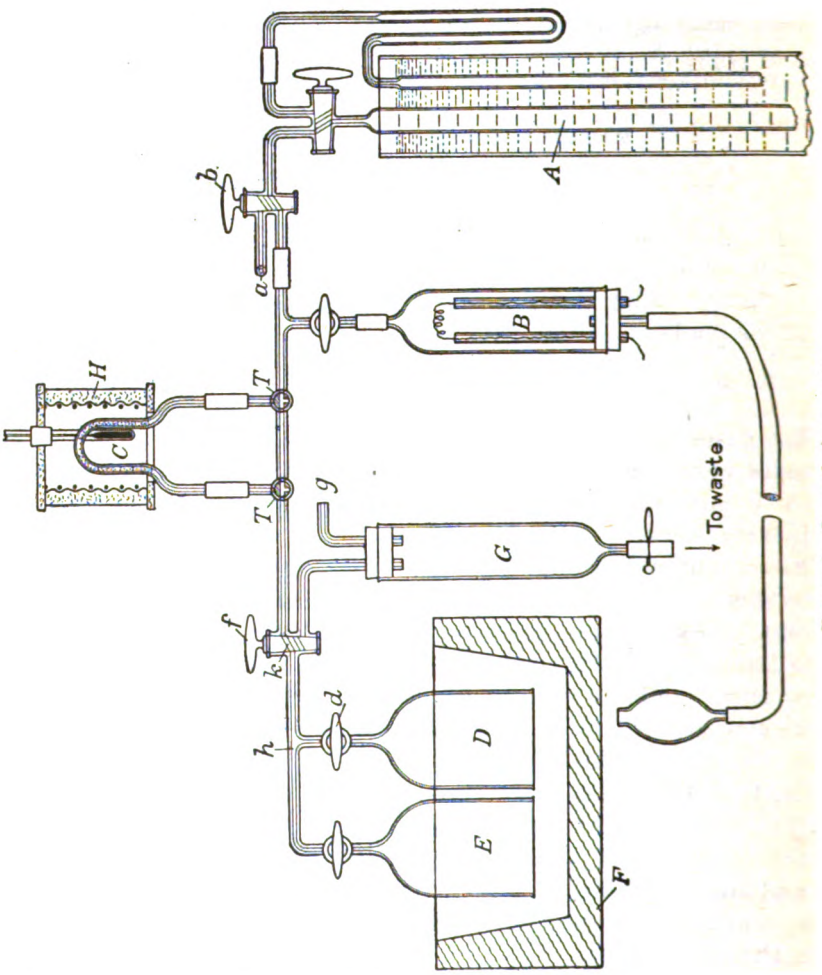

FIGURE 1.—Gas-analysis apparatus.

After freeing the copper oxide tube of oxygen by several passages of the gas through it into the pyrogallol, the carbon monoxide and hydrogen were burned together at 275° C. The contraction in volume was equal to the volume of hydrogen in the sample, and the CO_2 produced was calculated to CO. The residual gas, containing nothing but nitrogen and saturated hydrocarbons of the series C_nH_{2n+2}, was burned with oxygen in the slow-combustion pipette.

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Earnshaw's [a] method of calculation was used. The general equation for the combustion of hydrocarbons of the series C_nH_{2n+2} is:

$$C_nH_{2n+2}+\left(\frac{3n+1}{2}\right)O_2=nCO_2+(n+1)\,H_2O$$

If the volume of hydrocarbons be called V, the CO_2 produced A, and the contraction C, then

$$A=nV \text{ or } n=\frac{A}{V}$$

$$C=\left(V+\frac{3n+1}{2}V\right)-nV$$

$$C=V\left(\frac{n+3}{2}\right)$$

$$V=\frac{2C-A}{3}$$

It is impossible by simple combustion to determine the relative volumes of the various members of the series present, but the value of n as calculated is an index of the complexity and gives a general idea of the proportion of homologs higher than CH_4. If it be assumed that only CH_4 and C_2H_6 are present, then the value of n greater than unity is the proportion of C_2H_6—for example, $n=1.36$; then 36 per cent of the hydrocarbons is ethane and 64 per cent methane.

Nitrogen, being a minor constituent and its determination by difference subject to all the other errors in the analysis, has been omitted in the tables of gas yields but allowed for in calculating the weight of the gas.

The gas yields are expressed as cubic centimeters per 100 grams, and can be reduced to cubic feet per ton by dividing by 3.1.

DETERMINATION OF WATER.

Coal when heated gives up water in considerable quantity. Free surface water such as would be mechanically held by coal after being wetted was eliminated from the samples by air drying. Some of the inherent free water, which exists probably as one phase of a disperse system (colloidal), is also removed by air drying, but much of it remains in the coal. At the time the experiments were made, no method was known for determining with certainty the inherent water. The empirical method of drying the coal over phosphorus pentoxide in a vacuum desiccator was adopted, and the loss in weight designated as "moisture." This value was deducted from the total water produced during carbonization and the difference designated empirically "water of decomposition."

[a] Earnshaw, E. H., Water gas: Jour. Franklin Inst., vol. 146, September, 1898, pp. 161-176.

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The samples as used generally contained approximately the "moisture" content indicated in the table of analyses. If a different sample was used its moisture content was determined and the results all calculated on the basis of the moisture content indicated in the table.

In the carbonization experiments water was absorbed in phosphorus pentoxide contained in boats, the whole being weighed in stoppered tubes. Corrections were made for moisture absorbed during the transfer of the boat from the weighing tube to the apparatus, as determined by experiment.

Phosphorus pentoxide also absorbs ammonia, phenols, and nitrogen bases.[a] For this reason the results for water of decomposition are high, as no correction was made for this factor. Only traces of oils were found in any of the experiments. The magnitude of the error involved in the water determination could hardly have been as much as 1 per cent of the weight of the coal in any case, and usually was much less, considering the small quantities of reacting substances that reach the phosphorus pentoxide.[b]

DETERMINATION OF TAR AND OIL.

The tar and oil distilled from the coal were generally determined by deducting the sum of the weight of water and calculated weight of the gas from that of the total volatile matter. In some of the experiments the tar was determined by direct weighing. No attempt was made to determine its character or composition because of the small quantities obtained.

USE OF VACUUM.

In order to study the primary decomposition processes, the expedient of reduced pressure was employed in these experiments. By the use of a vacuum pump, the gases and vapors are quickly withdrawn from the heated retort and remain for a minimum time in contact with heated surfaces. The pressure on a system striving for chemical equilibrium at a given temperature, as in a retort where the coal and primary volatile products are reacting with each other, is an important factor in determining the character of the end products. If the distillation of the coal is carried out slowly enough so that secondary reactions may take place, which tends toward establishment of equilibrium, then the partial pressures of all the reacting substances and consequently the total pressure on the system are important factors in determining the reactions and the final end prod-

[a] Lambris, G., Wasserbestimmung, in den bei der Destillation von Brennstoffen auftretenden flüchtigen Producten: Ztschr. anorg. Chem., Bd. 81, 1913, pp. 24-39.

[b] Lambris, G., Loc. cit.

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ucts. If, however, the pressure be kept very low and the carbonization carried out rapidly, the liquid products distill quickly from the retort to cooler surfaces and the gases are withdrawn from the system before sufficient time has elapsed for secondary reactions to occur.

The rate at which volatile matter (other than water) is evolved from most coals is exceedingly slow up to a temperature of about 250° C. Although this temperature does not mark a transition temperature where volatile matter suddenly begins to evolve, it has been adopted as the lowest practicable temperature at which to begin experiments. To ascertain whether gases are evolved more rapidly in a partial vacuum, such as was used in the work described in this paper, than at atmospheric pressure or whether the temperature at which appreciable decomposition of the coal begins can be lowered in a vacuum, coals No. 650 and No. 43 were heated at 250° C. in an atmosphere of nitrogen at normal pressure. The apparatus, which is shown in figure 2, consisted of a bulb *a* for the coal, a bulb *b* for condensing water with a freezing mixture, a three-way cock *c*, and a large gas reservoir which could be used as a pump as well as for a gas receiver. The yields of gas were practically the same as those obtained under reduced pressure in experiments described in subsequent pages.

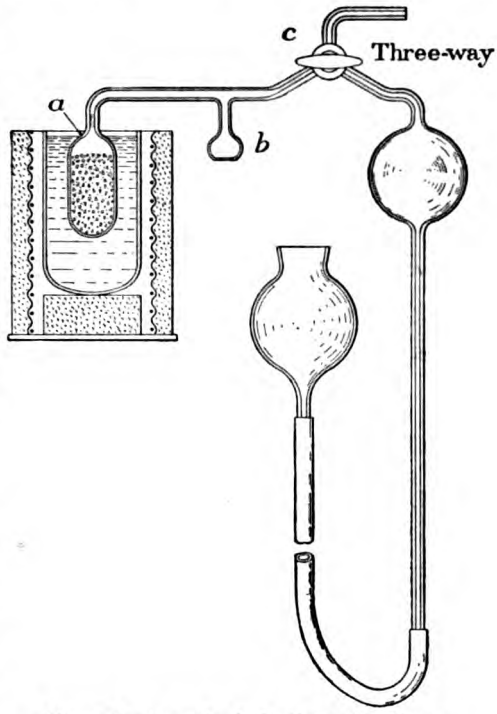

FIGURE 2.—Apparatus for heating coal in nitrogen.

DISTILLATIONS TO DETERMINE EASE OF DECOMPOSITION OF DIFFERENT COALS.

Coals vary greatly in their ease of decomposition. A thin layer of coal of 20 to 40 mesh size was placed in the annular space formed by two cylinders of fine platinum gauze (fig. 3). The diameter of the outer capsule was 10 mm. and that of the inner was 5 mm. A stiff platinum wire was bent into a ring, 20 mm. in diameter, around

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the capsule and served also as a handle. The capsule was hung from a fine platinum wire attached to a helical brass spring with a small piece of iron between. All these devices were contained in a long quartz tube 22 mm. in internal diameter, which was fixed in an upright position with its sealed end down and heated electrically to 1,000° C. By means of a solenoid acting on the iron, the capsule containing the coal was lowered into the heated zone for 5 seconds and then quickly jerked out by the spring. The tube was evacuated before each test. The average results from several concordant tests on 0.400 gram of each coal are given in the following table. In spite of the precautions taken to obtain a thin layer and heat it evenly, carbonization was not uniform throughout the coal mass. However, it is evident that coal No. 43 decomposed to a greater extent than the other two coals.

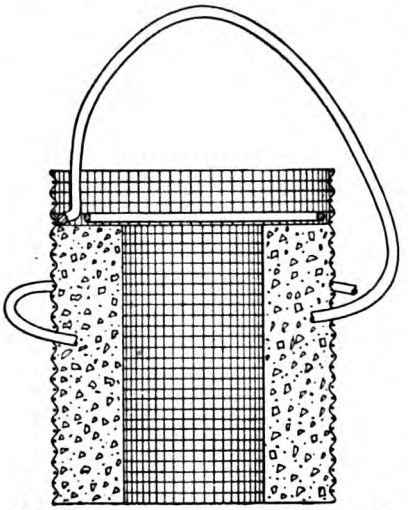

FIGURE 3.—Platinum-gauze capsule for quick heating of coal.

Volatile matter distilled in 5 seconds at 1,000° C. from 0.400 gram of coal.

Coal.	Gas yield.						Volatile.
	CO_2.	CO.	Unsaturated hydrocarbons.	C_nH_{2n+2}.	H_2.	Total.	
	C. c.	*C. c.*	*C. c.*	*C. c.*	*C. c.*	*C. c.*	*Per cent.*
New River (No. 674)	0.68	0.37	0.90	3.10	0.82	5.87	5.7
Pittsburgh (No. 650)	.33	.67	1.12	4.10	1.15	7.37	9.0
Wyoming (No. 43) [a]	3.37	4.17	2.65	5.05	2.42	17.66	36.3

[a] Not air-dried, approximately 22 per cent H_2O.

DISTILLATIONS OF VOLATILE MATTER AT TEMPERATURES BELOW 500° C.

PRELIMINARY EXPERIMENTS.

In order to gain some definite information on the theoretical effect of temperature, the coal should be heated instantaneously to the desired temperature in order that no volatile matter may be evolved at lower temperatures, but this does not appear to be possible experimentally.

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APPARATUS AND METHOD OF USE.

A sketch of an apparatus for heating coal very quickly is shown in figure 4. The retort was made of soft-glass tubing 22 mm. in internal diameter. The lower end was heated in a bath of fused sodium and potassium nitrates. The coal was contained in the feeding device in the upper part of the retort, which consisted of two short sections of glass tubing, the shorter one fitting closely but sliding easily in the other. The inner tube containing the coal was supported by a lead button and steel wire as shown. A small cylindrical roll of sheet iron was sprung tightly within it. On the outside of the retort was placed a coil of wire wound on glass, serving the double function of solenoid and mercury seal. When an electric current was passed through the solenoid, the sheet iron was acted upon magnetically and raised the coal container, allowing a few particles of coal to fall over the edge of the button into the bottom of the retort. It was possible to avoid dropping too much coal at once and also to feed it in as rapidly as was desired. The evolved gases were drawn off through a side tube over P_2O_5 by means of a mercury pump of the Töpler type. The mercury was raised and lowered in the pump by means of pressure and vacuum on the reservoir, and strokes could be made rather rapidly. Distillations were made with ease in this apparatus at 10 mm. pressure.

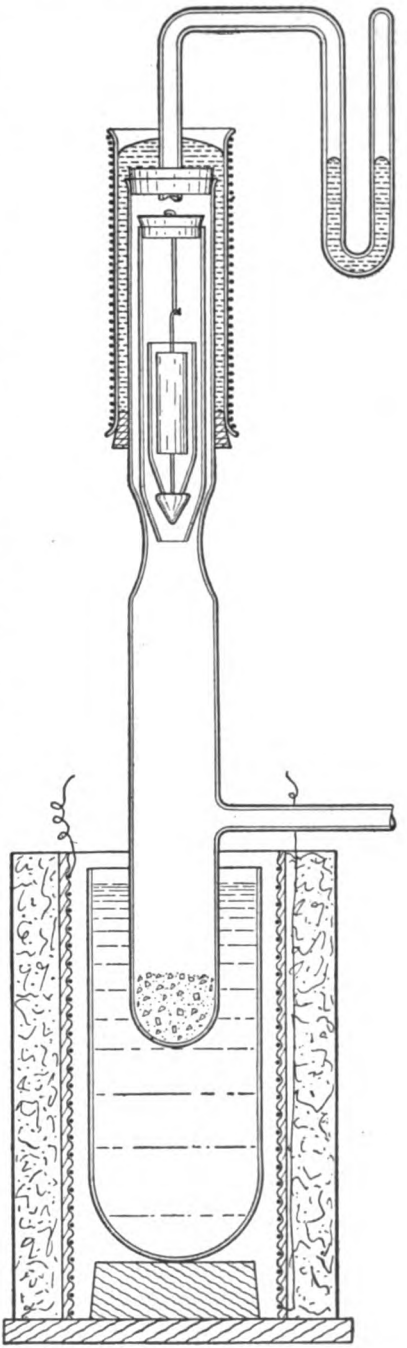

FIGURE 4.—Apparatus for rapidly heating coal under reduced pressure.

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RESULTS OF TESTS.

Results of tests at 475° C. are given in Table 1. At this temperature gas and tar were evolved quietly and most of the tar condensed in the delivery tube. At higher temperatures, however slow the rate of feed, tar distilled into the upper part of the retort and caused the particles of coal (40 to 60 mesh) to stick on the cold walls. For this reason results obtained in a hard-glass retort at 600° C. are not worth reporting. Another fact brought out in these preliminary experiments, which caused the abandonment of this type of apparatus, was the great effect of time on the yield of gases. Each of these experiments, reported in Table 1, lasted about 1½ hours, with the exception of No. 23, which lasted 3 hours. The experiment was stopped a few minutes after dropping in the last particles of coal. It is evident that the first particles of coal fed in were heated 1½ hours longer than the last particles and that the gas yields from them were very different. The time, then, for low-temperature work must be great enough to reduce such factors very materially or to eliminate them.

TABLE 1.—*Gases evolved from coals by rapid heating to 475° C.*

Item.	Pittsburgh (No. 650).	New River, W. Va. (No. 674).		Illinois (No. 48).	Wyoming (No. 43).
Experiment No.	18	20	23	24	17
Weight of coal.....grams.	2.0	2.5	1.8	2.0	2.0
Duration of test.....hours.	1½	1½	3	1½	1½
Total gas.....c. c. per 100 grams coal.	3,893	2,593	5,611	3,057	6,070
NH_3 and H_2S.....do....	160	33	30	57	124
CO_2.....do....	162	204	248	405	1,857
CO.....do....	160	82	177	305	1,400
Unsaturated hydrocarbons.....do....	337	232	358	275	342
C_nH_{2n+2}.....do....	2,720	1,780	3,588	1,645	1,817
Hydrogen.....do....	354	262	1,210	370	530
Value of n in C_nH_{2n+2}.....do....	1.35	1.28	1.24	1.48	1.32
Composition of gas:					
NH_3 and H_2S.....per cent.	4.10	1.25	.55	1.85	2.05
CO_2.....do....	4.10	7.85	4.40	13.25	30.50
CO.....do....	4.10	3.15	3.15	10.00	23.10
Unsaturated hydrocarbons.....do....	8.65	9.00	6.40	9.00	5.65
C_nH_{2n+2}.....do....	70.00	68.65	64.00	53.80	30.00
Hydrogen.....do....	9.05	10.10	21.50	12.10	8.70

DISTILLATIONS IN VACUUM.

Tests were then made by distilling the coal in a vacuum at temperatures below 500° C.

APPARATUS AND METHOD OF USE.

The apparatus used for these temperatures is sketched in figure 5. The coal was contained in a glass bulb, *a*, of such size that the charge (15 grams in most tests) occupied at least 75 per cent of its volume. The coal was air-dried and sized between 40 and 100 mesh. After charging, a small plug of asbestos was inserted in the neck of the bulb and the bulb sealed on the apparatus. Before beginning the

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heating, the atmosphere in the apparatus was exhausted by the mercury pump to less than 1 mm. pressure. Then the bulb was warmed a little and the nitrate bath, just melted at 220° C., was gradually raised till the bulb was submerged. More electric current was supplied to the furnace and the temperature raised to the degree desired as quickly as was consistent with keeping the pressure at 10 to 15 mm. With a rapidly decomposing coal like No. 43 the pressure was sometimes 25 mm. during the early part of the test. About one-half hour was required to bring the temperature to the required

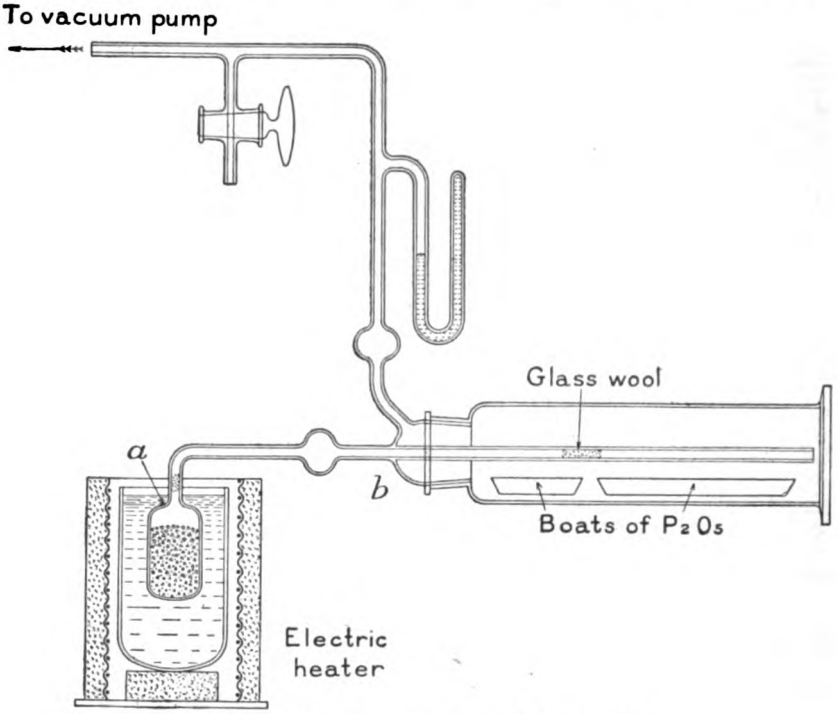

FIGURE 5.—Apparatus for distilling coal under reduced pressure.

point. The temperature of the retort walls was not at any point more than the temperature of the coal.

Most of the tests were run for a period not exceeding 13 hours, that is, during the time of the principal evolution of volatile matter. However, two coals were heated for long-continued periods (230 to 240 hours) in the apparatus shown in figure 6. The temperature of the nitrate bath was kept constant within $\pm 5°$ during most of the long run by the simple expedient of supplying gas at constant pressure to the burner.

A continuous record of the temperature was obtained by a Siemens and Halske recording millivoltmeter and thermoelement. The gas

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reservoir was large enough to prevent the pressure from rising above 40 mm. and was exhausted at least once every 24 hours. The mercury trap connecting with the pump made stopcocks unnecessary and assured that no air entered the apparatus.

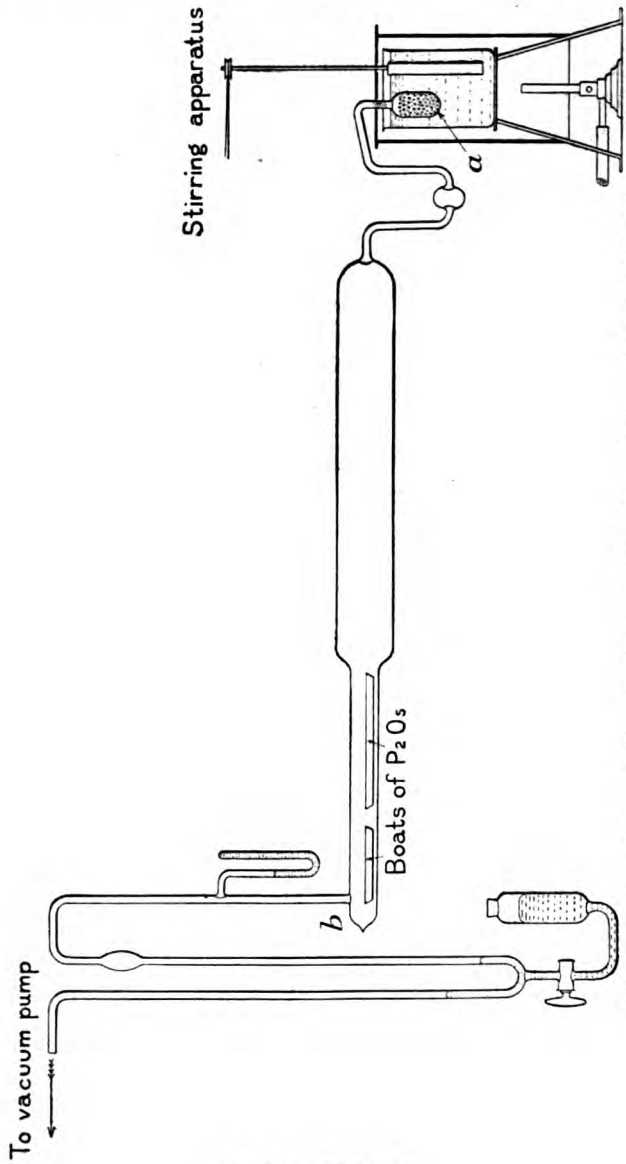

FIGURE 6.—Apparatus for continuous distillation of coal under reduced pressure for long periods of time.

RESULTS OF TESTS.

The results are presented in Tables 2 to 7. Tables 2 and 3 give the results of heating the four coals to 250° and 350° C., respectively. Tables 4 and 5 show the detailed results of the several fractions collected

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at 350° C. for Pittsburgh coal (No. 650) and Wyoming coal (No. 43). Table 6 shows the results of heating the four coals to a temperature of 450° C. Table 7 shows the results of an experiment (run 78) in which the residue from run No. 74 (coal No. 650) was further heated for 191 hours at 450° C.

TABLE 2.—*Products of heating coal at 250° C. (482° F.).*

Item.	Pittsburgh (No. 650).	New River, W. Va. (No. 674).	Illinois (No. 48).	Wyoming (No. 43).
Experiment No	60	61	62	65
Weight of coal (air dry) grams	15.0	15.0	15.0	15.0
Duration of test hours	7.0	6.5	6.75	7.0
Total volatile matter (without moisture) per cent	.73	.66	2.61	4.69
Tar do	.13	.04	.32	(a)
Water of decomposition do	.50	.50	1.95	3.65
Total gas do	.10	.12	.35	1.04
Total gas c. c. per 100 grams coal	57	110	267	659
NH_3 and H_2S do		6	21	23
CO_2 do	34	42	156	478
CO do	6	12	35	81
Unsaturated hydrocarbons do	1	1	2	6
Saturated hydrocarbons (C_nH_{2n+2}) do	3	5	6	21
Hydrogen do	0			
Value of n in C_nH_{2n+2}			1.53	1.25
Composition of gas:				
NH_3 and H_2S per cent		5.5	7.9	3.5
CO_2 do	60.0	38.1	58.9	72.4
CO do	10.5	11.0	12.8	12.4
Unsaturated hydrocarbons do	1.9	.9	.8	.9
Saturated hydrocarbons do	5.3	4.5	2.2	3.2
Hydrogen do	0.0			

[a] Trace of colorless oil.

TABLE 3.—*Products of heating coal at 350° C. (662° F.).*

Item.	Pittsburgh (No. 650).		New River (No. 674).	Illinois (No. 48).	Wyoming (No. 43).		
Experiment No	63	74 [a]	69	66	67	72 [a]	188 [b]
Weight of coal (air-dry) grams	15.0	15.0	15.0	15.0	15.0	15.0	15.0
Duration of test hours	13	239	7	13.5	7	234	13
Total volatile (without moisture) per cent	5.90	12.93	1.87	7.95	15.0	22.97	16.74
Tar do	4.28	8.15	.66	3.08	5.52	5.92	5.53
Water of decomposition do	1.12	2.94	.88	3.68	6.32	11.07	6.64
Total gas do	.50	1.84	.33	1.19	3.16	5.98	4.57
Total gas c. c. per 100 grams coal	[c] 449	1,891	256	937	2,342	4,890	3,215
NH_3 and H_2S do	21	111	15	37	232	234	240
CO_2 do	73	117	104	336	1,142	1,812	1,715
CO do	23	71	24	87	358	780	577
Unsaturated hydrocarbons do	44	196	8	67	107	286	123
Saturated hydrocarbons (C_nH_{2n+2}) do	229	1,235	41	307	367	1,287	494
Hydrogen do	20	138	9	32	19	287	66
Value of n in C_nH_{n+2}	1.57	1.52	1.75	1.60	1.49	1.46	1.55
Composition of gas:							
NH_3 and H_2S per cent	4.7	5.8	5.9	4.0	9.9	7.0	7.50
CO_2 do	16.2	6.2	40.1	35.7	48.7	37.8	53.35
CO do	5.1	4.1	9.1	9.3	15.3	16.3	17.95
Unsaturated hydrocarbons do	9.8	10.6	3.1	7.2	4.6	6.0	3.80
Saturated hydrocarbons do	51.0	65.9	16.0	32.7	15.7	26.9	15.35
Hydrogen do	4.5	7.4	3.5	3.4	.8	6.0	2.05

[a] Continuous long-time test; for detailed data of successive gas fractions see Tables 4 and 5, p. 20.
[b] In the first 6½ hours of this test 271 c. c. of gas was obtained, of which 24.2 per cent was CO_2, 7.2 per cent CO, and 2.5 per cent hydrogen.
[c] This test was made one year later on a different sample of the same coal.

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TABLE 4.—*Products of continuous long-time heating of Pittsburgh coal (No. 650) at 350° C. (662° F.).*

(Experiment No. 74.)

Item.	A	B	C	D	E	F	G	Total.
Duration of test.....hours..	1	22	32	40	27	45	72	239
Mean temperature.....°C..	25 to 350	354	353	349	351	350	351	.....
Gas yield.....c. c. per 100 grams coal..	113	655	371	229	124	157	[a] 242	1,891
NH_3 and H_2S.....do....	13	39	19	11	16		13	111
CO_2.....do....	53	38	9	5	5		5	115
CO.....do....	47		8	5	5		5	70
Unsaturated hydrocarbons.....do....	75		42	25	30		25	197
Saturated hydrocarbons (C_nH_{2n+2}) .....c. c. per 100 grams coal..	445		264	167	201		158	1,235
Hydrogen.....do....	42		27	17	23		29	138
Value of n in C_nH_{2n+2}	1.62		1.51	1.47	1.45		1.42	.....
Composition of gas:								
NH_3 and H_2S.....per cent..	11.2	6.0	5.20	4.65	5.60		5.35	5.80
CO_2.....do....	47.3	5.8	2.55	2.20	1.80		2.20	6.20
CO.....do....	6.12		2.15	2.30	1.80		2.20	4.10
Unsaturated hydrocarbons...do....	9.71		11.35	10.75	10.55		10.45	10.60
Saturated hydrocarbons.....do....	57.84		71.20	72.90	71.45		65.10	65.85
Hydrogen.....do....	5.45		7.10	7.30	8.20		12.10	7.45

[a] Rate remained uniform during last 117 hours, at about 3.4 c. c. per hour per 100 grams.

TABLE 5.—*Products of continuous long-time heating of Wyoming coal (No. 43) at 350° C. (662° F.).*

(Experiment No. 72.)

Item.	A	B	C	D	E	F	G	Total.
Duration.....hours..	1	22	31	41	24	26	89	234
Mean temperature.....°C..	25 to 350	346	342	348	351	346	350	.....
Gas yield....c. c. per 100 grams coal..	1,539	1,734	442	449	221	144	[a] 294	[a] 4,890
NH_3 and H_2S.....do....	98	173	30	16	7		11	334
CO_2.....do....	1,030	542	75	71	53		42	1,812
CO.....do....	244	302	67	68	53		47	780
Unsaturated hydrocarbons.....do....	35	132	43	39	22		15	286
Saturated hydrocarbons (C_nH_{2n+2}) .....c. c. per 100 grams coal..	103	487	177	193	167		158	1,287
Hydrogen.....do....	5	49	40	54	60		79	287
Value of n in C_nH_{2n+2}	1.49	1.59	1.49	1.39	1.30		1.26	.....
Composition of gas:								
NH_3 and H_2S.....per cent..	6.40	9.95	6.70	3.55	1.95		2.95	6.9
CO_2.....do....	66.90	31.25	16.90	15.90	14.40		11.65	37.1
CO.....do....	15.85	17.40	15.10	15.15	14.45		13.10	16.0
Unsaturated hydrocarbons.do...	2.30	7.60	9.80	8.60	5.90		4.15	6.0
Saturated hydrocarbons...do....	6.80	28.05	40.00	43.00	45.80		43.80	26.3
Hydrogen.....do....	.30	2.85	9.05	12.00	16.50		21.80	6.0

[a] The gas obtained during the last 15 hours of the run was lost by accident; for the preceding 24 hours the rate had been about 4 c. c. per hour.

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TABLE 6.—*Products of heating coal at 450° C. (842° F.).*

Item.	Pittsburgh (No. 650).	New River, W. Va. (No. 674).	Illinois. (No. 48).	Wyoming (No. 43).	
Experiment No.	73	71	75	76	[c]194
Duration.....hours.	8.5	9.0	9.5	10.0	7.0
Weight of coal, air-dry.....grams.	5.0	5.0	5.0	5.0	4.0
Total volatile, without moisture.....per cent.	21.07	12.18	20.82	29.47	26.20
Tar.....do.	12.76	6.08	7.73	8.17	6.68
Water of decomposition.....do.	4.30	2.30	8.29	12.64	11.20
Gas yield.....do.	4.01	3.80	4.80	8.66	8.32
Total gas yield.....c. c. per 100 grams coal.	4,694	5,498	5,621	8,194	7,263
Gas in first 3 hours of run.....do.	[a] 4,385	4,410	4,370	[b] 7,426	.....
NH_3 and H_2S.....do.	138	48	104	432	359
CO_2.....do.	174	223	533	2,128	2,318
CO.....do.	187	130	483	1,426	1,183
Unsaturated hydrocarbons.....do.	352	334	320	394	237
Saturated hydrocarbons (C_nH_{2n+2}).....do.	3,100	3,551	3,158	2,647	2,234
Hydrogen.....do.	653	1,168	935	1,129	932
Value of n in C_nH_{2n+2}	1.37	1.21	1.30	1.31	1.36
Composition of gas:					
NH_3 and H_2S.....per cent.	2.94	.87	1.85	5.27	4.95
CO_2.....do.	3.71	4.05	9.48	25.95	31.90
CO.....do.	4.00	2.36	8.59	17.40	16.30
Unsaturated hydrocarbons.....do.	7.50	6.07	5.70	4.79	3.25
Saturated hydrocarbons.....do.	66.00	64.56	56.18	32.28	30.80
Hydrogen.....do.	13.98	21.24	16.64	13.76	12.80

[a] First 6 hours. [b] First 5 hours. [c] See footnote, Table 3, experiment 188.

TABLE 7.—*Products of continuous long-time heating of Pittsburgh coal (residue from experiment No. 74) at 450° C.*

(Experiment No. 78.)

	A	B	C	D	E	F	G	H	I	Total.
Duration.....hours.	1	4	20	24	30	40	24	25	23	191
Mean temperature.....°C.	350–450	450	454	455	456	457	450	465	452	.....
Total gas yield, c. c. per 100 grams coal	838	2,565	1,862	650	483	374	147	227	119	7,265
NH_3 and H_2S..c. c. 100 grams coal	25	83	202	14	6	3		3		336
CO_2.....do.	27	33	49	20	13	7		8		157
CO.....do.	27	97	75	39	30	27		38		333
Unsaturated hydrocarbons, c. c. per 100 grams coal.	68	92	18	1	1	1		2		183
C_nH_{2n+2}.....do.	604	1,630	770	267	166	123		162		3,722
H_2.....do.	87	630	748	309	267	213		280		2,534
Value of n in C_nH_{2n+2}	1.34	1.15	1.04	1.01	1.00	1.00		1.00		.....
Composition of gas:										
NH_3 and H_2S..per cent.	3.00	3.20	10.85	2.15	1.25	.80		.60		4.65
CO_2.....do.	3.25	1.30	2.60	3.10	2.70	1.85		1.60		2.15
CO.....do.	3.25	3.80	4.00	6.00	6.20	7.20		7.70		4.60
Unsaturated hydrocarbons.....per cent.	8.10	3.60	.95	.15	.20	.25		.40		2.50
C_nH_{2n+2}.....do.	72.10	63.60	41.40	41.10	34.35	32.90		32.85		51.20
H_2.....do.	10.30	24.50	40.20	47.50	55.30	57.00		56.85		34.90

NOTE.—Total volatile, 8.78 per cent, as follows: H_2O, 2.97 per cent; gas, 4.42 per cent; and tar, 1.39 per cent.

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DISTILLATIONS OF VOLATILE MATTER AT TEMPERATURES ABOVE 500° C.

VACUUM DISTILLATION AT 600° C.

Distillations at a temperature of 600° C. were made in a manner similar to those made at temperatures below 500° C., except that the coal was contained in a horizontal hard-glass bulb heated in air by means of an electric furnace. The thermoelement junction was placed directly on the outside of the retort wall. The results of these experiments are given in Table 8.

TABLE 8.—*Products of heating coal at 600° C.*

Item.	Pittsburgh (No. 650).	New River, W. Va. (No. 674).	Illinois (No. 48).	Wyoming (No. 43).
Experiment No	191	190	192	193
Weight of coal, air-dry.....grams	2.000	2.000	2.000	2.000
Duration of test.....hours	6.0	6.0	6.0	6.0
Total volatile (without moisture).....per cent	26.32	17.92	26.74	32.65
Tar.....do	13.29	7.59	8.16	5.93
Water of decomposition.....do	6.47	3.31	9.78	13.55
Total gas.....do	6.56	7.02	8.80	13.17
Total gas.....c. c. per 100 grams coal	11,790	13,865	15,730	17,930
NH_3 and H_2S.....do	340	170	273	535
CO_2.....do	270	440	883	3,130
CO.....do	755	608	1,882	2,910
Unsaturated hydrocarbons.....do	390	452	362	355
C_nH_{2n+2}.....do	4,765	5,700	5,270	4,135
Hydrogen.....do	5,270	6,495	7,060	6,865
Value of n in C_nH_{2n+2}	1.26	1.18	1.20	1.20
Composition of gas:				
NH_3 and H_2S.....per cent	2.90	1.20	1.75	3.00
CO_2.....do	2.30	3.20	5.60	17.45
CO.....do	6.40	4.40	11.95	16.25
Unsaturated hydrocarbons.....do	3.30	3.25	2.30	2.00
C_nH_{2n+2}.....do	40.40	41.15	33.50	23.05
Hydrogen.....do	44.70	46.80	44.90	38.25

VACUUM DISTILLATION AT 1,050° C.

Some distillations were then made in which the coal was heated to 1,050° C. The retort used for distilling coal at high temperatures, with a minimum amount of secondary decomposition, was a clear quartz tube that was 22 millimeters in internal diameter and was closed at one end. The coal was contained in a small platinum crucible with a cap cover, supported in the inverted tube on a quartz rod as shown in figure 7. The quartz tube was connected to the pump by means of a rubber stopper and mercury seal; the gases were passed over phosphorus pentoxide. The wires of the thermocouple passed in from below, with the hot junction directly against the side of the crucible. The temperature of the outside wall of the quartz tube was also taken and it was found that the temperature inside the tube followed that of the wall closely.

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In order to minimize carbonization of tar, the cold electric heater was so placed over the retort that the tube extended about 8 centimeters within the heating coil. The coal was then heated up slowly, in about 45 minutes, to a temperature of 600° C. The tar drained out of the heated space as fast as formed, the heavier parts condensing on the walls just outside the heater. The heater was then lifted about 3 centimeters, which effectually prevented reheating the tar condensed; the temperature was raised to 1,050° C. in 15 minutes and the distillation stopped. Coal No. 674 was the only coal that was still giving off gas at an appreciable rate at 1,050° C. In

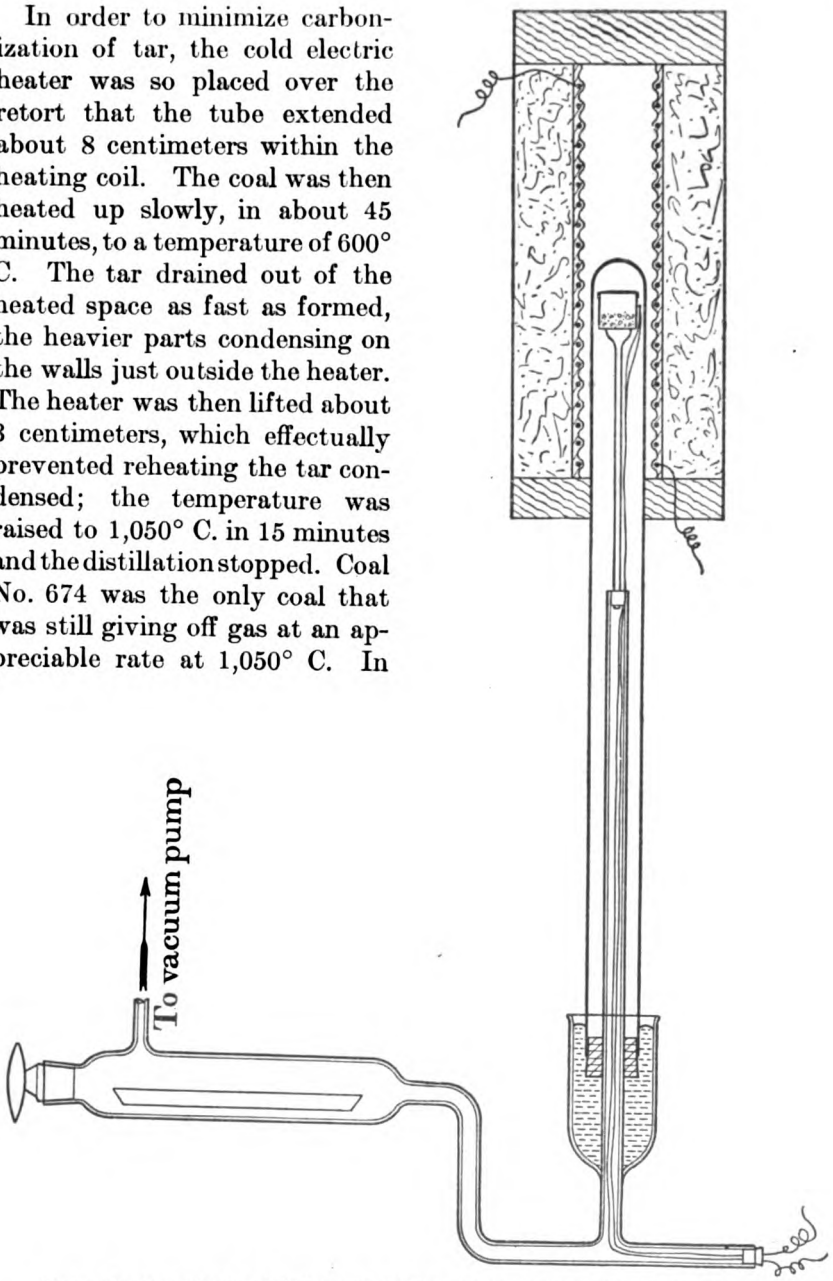

FIGURE 7.—Apparatus for distilling coal at high temperatures and under reduced pressure.

these tests there was never the slightest deposit of free carbon on the crucible or retort walls. The pressure in the apparatus was 20 to 25 millimeters. The results of the tests are given in Table 9.

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TABLE 9.—*Products of heating coal to 1,050° C.*

Item.	Pittsburgh (No. 650).	New River, W. Va. (No. 674).	Illinois (No. 48).	Wyoming (No. 43).
Experiment No	92	94	93	95
Weight of coal, air-dry, grams	1.000	1.000	1.000	0.500
Duration of test, hours	1.0	1.0	1.0	1.0
Total volatile (without moisture), per cent	33.7	21.7	32.0	40.5
Tar and loss, do	18.9	9.0	12.2	10.5
Water of decomposition, do	5.5	3.4	8.2	14.3
Total gas, do	9.3	9.3	11.6	15.6
Total gas, c. c. per 100 grams coal	27,750	35,935	30,515	30,583
NH_3 and H_2S, do	425	145	280	177
CO_2, do	425	490	990	2,963
CO, do	1,375	1,265	2,998	5,355
Unsaturated hydrocarbons, do	510	415	510	463
C_nH_{2n+2}, do	5,565	6,130	5,217	3,745
Hydrogen, do	19,450	27,490	20,520	17,880
Value of n in C_nH_{2n+2}	1.20	1.15	1.19	1.17
Composition of gas:				
NH_3 and H_2S, per cent	1.50	.40	.90	.60
CO_2, do	1.50	1.35	3.25	9.70
CO, do	5.00	3.50	9.80	17.50
Unsaturated hydrocarbons, do	1.85	1.15	1.65	1.50
C_nH_{2n+2}, do	20.05	17.05	17.10	12.20
Hydrogen, do	70.10	76.55	67.30	58.50

EFFECT OF RAPID AND SLOW HEATING.

RESULTS OF HEATING ORIGINAL COAL.

The results obtained by heating at different rates 1 gram of Pittsburgh coal in the apparatus shown in figure 7 are given in Table 10, following. Experiments 87 and 88 were made at reduced pressure, as was experiment 92. In experiments 89, 90, and 91 the furnace was heated to 1,050° C. and dropped over the quartz tube. The sudden evolution of gas made it impossible to keep a reduced pressure, but a cylinder of 750 c. c. capacity interposed between the retort and the pump gave a total volume, with that of the pump, of at least 1,500 c. c., so that relatively a very small portion of the gas remained in the heated retort at any period of the experiment. The gas was withdrawn as fast as possible, but reached a pressure of several centimeters. In experiments 87, 88, and 89 the coal was put into an annular space 2 mm. thick, formed by inserting a cylinder of platinum foil in the crucible, with the object of obtaining more even heating. No carbon was deposited in tests 87 and 88, but in the other tests the retort walls and crucible were black with it. The differences in gas yield in the cases of more rapid heating probably came largely from secondary decomposition of tar. The higher total volatile matter may have been due to particles carried out of the crucible and lost in the rapid evolution of gases, but no evidence of such loss appeared in run 90.

The tar yields as indicated by difference, after weighing the residue and water and calculating the weight of gas, seem abnormally high,

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and not much reliance should be placed on this part of the data. One source of error may lie in the free carbon formed at high temperatures, and counted here as tar.

TABLE 10.—*Results of rapid and slow heating of Pittsburgh coal (No. 650).*

Item.	Results of experiment No. —				
	87	88	89	90	91
Maximum temperature....°C..	1,050	1,060	1,060	1,060	1,100
Time of heating coal to 600° C....minutes..	90	80	½	¼	½
Time 1,000° C....do....	270	190	6	2	7
Total time....do....	335	210	45	30	60
Total volatile....per cent..	34.7	34.6	39.5	36.8	38.8
Tar and loss....do....	18.1	18.9	.....	.....	.....
Total water....do....	6.3	6.5	5.3	4.8	5.6
Total gas....do....	10.3	9.3	12.0	.....	12.3
Total gas....c. c. per 100 grams coal..	29,570	28,480	29,570	.....	30,750
NH_3 and H_2S....do....	440	350	390	.....	420
CO_2....do....	490	520	495	.....	560
CO....do....	1,920	1,700	2,150	.....	2,140
Unsaturated hydrocarbons....do....	710	520	1,505	.....	1,610
CnH_{2n+2}....do....	5,570	5,550	6,400	.....	6,360
Hydrogen....do....	20,440	19,840	18,630	.....	19,660
Value of n in CnH_{2n+2}....do....	1.21	1.19	1.20	.....	1.20
Composition of gas:					
NH_3 and H_2S....per cent..	1.50	1.25	1.30	.....	1.35
CO_2....do....	1.65	1.80	1.65	.....	1.80
CO....do....	6.50	6.00	7.25	.....	6.95
Unsaturated hydrocarbons....do....	2.40	1.80	5.10	.....	5.20
CnH_{2n+2}....do....	18.85	19.45	21.60	.....	20.70
Hydrogen....do....	69.10	69.70	63.10	.....	64.00

RESULTS OF REHEATING RESIDUE.

The residue (test 78) from Pittsburgh coal No. 650 that had been heated 239 hours at 350° C. and 191 hours at 450° C. was further distilled in the apparatus shown in figure 7 for about 2 hours at 1,050° C., with the following results:

Results of heating residue to 1,050° C. for 2 hours.

Product.	Yield, based on residue.	Yield, based on original coal.	Proportion of total gas.
	C. c. per 100 grams.	*C. c. per 100 grams.*	*Per cent.*
CO_2	273	211	1.10
CO	1,850	1,430	7.50
Unsaturated hydrocarbons	24	18	.10
CH_4	780	603	3.15
H_2	21,680	16,750	88.15
Total gas	24,607	19,012	100.00
	Per cent.	*Per cent.*	
Total volatile	6.30	4.87	.....
Total gas yield	5.38	4.16	.....
Total H_2O	.82	.63	.....
Tar (by difference)	.10	.08	.....

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SECONDARY THERMAL REACTIONS AMONG VOLATILE PRODUCTS.

That the character of the volatile matter may be radically altered by superheating or passage over hot surfaces was demonstrated qualitatively in the following experiments:

REACTIONS AMONG GASES.

First, the effect of superheating the gases alone was investigated. Gas obtained by heating West Virginia coal (No. 674) at 475° C. and saturated with water vapor was passed back and forth 10 times through a quartz tube 25 cm. long and 2 mm. in internal diameter, heated for about 1 cm. of its length to dull red by a gas flame. A portion of the same gas was passed into the combustion pipette of the gas-analysis apparatus and the platinum wire made to glow for 2 minutes. The results of the experiments were as follows:

Results of tests with quartz tube and platinum wire.

	Original volume.	Volume obtained from quartz-tube test.	Volume obtained from glowing-wire test.
	C. c.	*C. c.*	*C. c.*
Total volume	100.00	116.05	122.70
NH_3 and H_2S	.45	.30	.00
CO_2	2.40	2.50	2.70
CO	1.35	2.20	1.70
Unsaturated hydrocarbons	7.10	15.80	18.25
C_nH_{2n+2}	[a] 69.65	[b] 67.15	[c] 64.85
H_2	17.75	27.10	34.00
O_2	.30	.00	.15
N_2	1.00	1.00	1.05

[a] $n=1.35$. [b] $n=1.07$. [c] $n=1.06$.

With the same quartz tube heated electrically for 10 cm. of its length, gas at atmospheric pressure and saturated with water vapor at room conditions was passed back and forth through the tube 10 times in about 10 minutes. The results, which are given in Table 11, following, show that the chief change in the gas is a cracking of the heavier saturated hydrocarbons.

TABLE 11.—*Results of experiments to determine secondary reactions among gases.*

	Gas distilled from Pittsburgh coal (No. 650) at 450° C.		Gas distilled from Pittsburgh coal (No. 650) at 350° C.		Gas distilled from Illinois coal (No. 48) at 450° C.		
	Original volume.	Volume after superheating, 650° C.	Original volume.	Volume after superheating, 650° C.	Original volume.	Volume after superheating, 750° C.	Volume after superheating, 800° C.
Total volume..cu. centimeters..	100.00	100.40	100.00	105.30	100.00	108.60	113.90
NH_3 and H_2S.....do....	2.95	0.00	5.60	5.10	1.80	1.10	.75
CO_2.....do....	3.70	3.70	5.40	5.95	11.50	11.55	11.55
CO.....do....	3.90	3.90	6.60	6.40	8.35	8.60	9.55
Unsaturated hydrocarbons.....cu. centimeters..	7.50	8.35	10.90	14.35	6.60	11.55	15.40
C_nH_{2n+2}.....do....	66.05	67.40	63.30	64.90	58.05	61.60	57.15
H_2.....do....	13.90	14.80	6.00	6.85	11.85	11.95	17.65
O_2.....do....	.40	.00	.50	.10	.20	.00	.10
N_2.....do....	1.60	2.25	1.70	1.65	1.65	2.25	1.75
Value of n	1.37	1.33	1.62	1.47	1.37	1.22	1.08

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REACTIONS AMONG GASES AND TAR VAPORS.

Much greater in extent was the secondary thermal decomposition of tar vapors and liquids as shown by subjecting the entire mixture of volatile products to superheating temperatures.

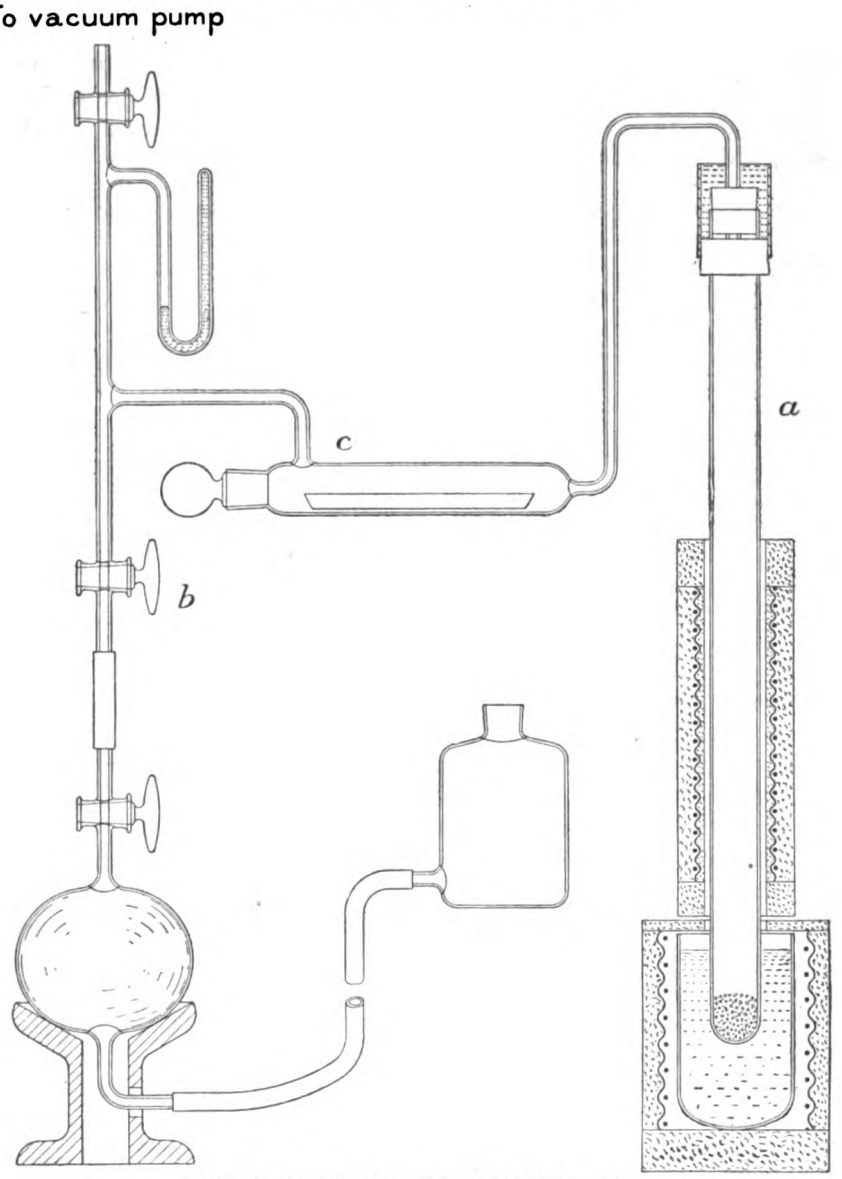

FIGURE 8.—Apparatus for superheating volatile matter.

PRELIMINARY TESTS.

In the preliminary experiments on superheating the volatile matter the apparatus shown in figure 8 was employed. In the bottom of

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the quartz tube *a*, which was 22 mm. in diameter and 375 mm. long, was placed 3 grams of Pittsburgh coal (No. 650). The coal was heated two hours at a temperature of 450° C. by means of the nitrate bath, while that part of the tube above the coal was heated for a length of 150 mm. to 750° C. Tests were carried out both at reduced and atmospheric pressure; in either case the apparatus was evacuated at the start. In the experiment at normal pressure, when enough gas had been evolved to fill the apparatus the stopcock *b* was opened and the gas collected in the container over mercury. Only gas yields were determined. The results were as follows:

Results of preliminary experiments in superheating volatile matter.

Product.	Yield per hundred grams of coal.		
	In vacuum, without superheating.	In vacuum, superheated to 750° C.	At normal pressure, superheated to 750° C.
	C. c.	C. c.	C. c.
NH_3 and H_2S	160	220	155
CO_2	160	138	253
CO	127	480	687
Unsaturated hydrocarbons	335	1,287	857
C_nH_{2n+2}	[a] 2,287	[b] 3,450	[c] 5,253
H_2	207	1,063	2,007
Total	3,276	6,638	9,212

[a] $n=1.51$. [b] $n=1.43$. [c] $n=1.05$.

TESTS WITH SHORTER SUPERHEATING FURNACE.

Further experiments were made in which the apparatus shown in figure 8 was slightly rearranged by placing the gas-collecting reservoir on the right, without the drying tube, and connecting it with the quartz tube by means of a T stopcock. The quartz tube could then be connected to the reservoir in tests at normal pressure, or to the pump through a drying tube in the tests made in vacuum. The experiments were made as before except that a shorter superheating furnace was used, only 60 mm. of the tube being heated. In some experiments broken pieces of fire brick were piled in on top of the coal, filling the tube above the heated zone, in order to increase the surfaces of contact. In each test 5 grams of Pittsburgh coal (No. 650) was used. This coal swells so badly at normal pressure that no test could be made without the fire brick to hold it down in the retort, although it did not swell when heated at a reduced pressure of 5 to 40 mm. The results are given in Table 12. The tests made at low pressure show how easily the decomposition of tar vapors into permanent gases may take place. The superheated zone was very short and gases and lighter oils were removed from the zone quickly under the conditions of the experiment,

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TABLE 12.—*Results of tests to determine secondary thermal decomposition of volatile matter evolved at 475° C. from coal.*

[Pittsburgh coal (No. 650) was used in these tests.]

Item.	Results, with conditions as stated, from experiment No.—					
	113 Atmospheric pressure; brick column; not superheated.	115 Atmospheric pressure; brick column in lower retort; not superheated.	118 Atmospheric pressure; brick column; superheated to 550° C.	116 Atmospheric pressure; brick column; superheated to 650° C.	121 15 to 40 mm. pressure; no brick; not superheated.	122 15 to 40 mm. pressure; no brick; not superheated.
NH_3 and H_2S.c.c. per 100 grams	182	157	162	206	73	136
CO_2.....do....	194	176	178	243	177	200
CO.....do....	174	150	120	316	203	203
Unsaturated hydrocarbons...c. c. per 100 grams..	298	298	378	1,064	403	390
C_nH_{2n+2}.....do....	2,860	3,050	3,032	4,571	3,732	3,660
H_2.....do....	206	268	262	960	586	623
Total.....do....	3,914	4,099	4,132	7,360	5,174	5,212
Value of n.....	1.44	1.40	1.39	1.33	1.35	1.35

Item.	Results, with conditions as stated, from experiment No.—					
	120 15 to 40 mm. pressure; no brick; superheated to 550° C.	124 15 to 40 mm. pressure; no brick; superheated to 650° C.	119 15 to 40 mm. pressure; brick column; superheated to 650° C.	123 15 to 40 mm. pressure; brick column; superheated to 650° C.	125 15 to 40 mm. pressure; no brick; superheated to 800° C.	126 15 to 40 mm. pressure; brick column; superheated to 800° C.
NH_3 and H_2S.c.c. per 100 grams	172	132	223	174	148	190
CO_2.....do....	192	198	190	209	256	240
CO.....do....	226	237	244	209	762	920
Unsaturated hydrocarbons...c. c. per 100 grams..	447	711	562	566	2,010	1,930
C_nH_{2n+2}.....do....	3,870	4,056	3,368	3,545	5,006	4,956
H_2.....do....	722	630	733	721	1,680	3,672
Total.....do....	5,629	5,964	5,320	5,424	9,862	11,908
Value of n.....	1.40	1.33	1.37	1.37	1.20	1.24

EFFECT OF SMALL HIGHLY HEATED SURFACE.

The effect of a small but highly heated surface on the volatile products was tried by distilling Pittsburgh coal (No. 650) in a glass bulb at 450° C. and passing the gases and tar vapors over a glowing platinum wire, 15 cm. of No. 22 wire made into a coil 3 cm. long being used. In the results designated as "normal yield" the wire coil was not heated. The results of the tests, which were made at atmospheric pressure, were as shown in the table following.

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Results of passing volatile products over a heated wire at atmospheric pressure.

	Experiment 107, normal yield.	Experiment 109, gases passed over heated wire.
NH_3 and H_2S.....c. c. per 100 grams coal..	202	271
CO_2.....do....	156	210
CO.....do....	136	344
Unsaturated hydrocarbons.....do....	237	1,340
C_nH_{2n+2}.....do....	2,540	3,650
H_2.....do....	241	1,285
Total.....do....	3,512	7,100
Value of n.....	1.44	1.16

CATALYTIC ACTION OF PLATINUM.

In this connection it is desirable to present some data, obtained in distilling coal in the apparatus shown in figure 7, on the catalytic effect of platinum on secondary reactions, which showed that a platinum container for the coal had no apparent effect. In experiments 52 and 56 the coal was contained in a platinum-gauze capsule; in experiments 55 and 42 the coal was contained in quartz capsules with asbestos-fiber covers. The experiments also show the effect of rapid and slow heating of the coal. In experiments 42 and 52 the coal was heated to 1,000° C. in 1¼ hours and was kept at that temperature 1 hour longer. In experiments 55 and 56 the capsule was attached to a quartz rod and raised into the hot retort by lifting with a magnet a piece of iron on the lower end of the rod; the distillation was continued 5 minutes and then the capsule withdrawn. The results of experiments made at reduced pressure were as follows:

Results of heating coal at reduced pressure in quartz and platinum crucibles.

[0.30 gram of Pittsburgh coal (No. 650) used in each test.]

Item.	Yield obtained by—			
	Slow heating.		Rapid heating.	
	Experiment 42, quartz crucible.	Experiment 52, platinum crucible.	Experiment 55, quartz crucible.	Experiment 56, platinum crucible.
CO_2.....cubic centimeters..	0.90	0.95	0.85	0.80
CO.....do....	4.90	5.40	7.20	6.65
Unsaturated hydrocarbons.....do....	1.35	1.40	6.70	6.80
C_nH_{2n+2}.....do....	16.35	14.80	19.40	19.50
H_2.....do....	48.75	50.90	47.30	46.20
Total.....do....	72.25	73.45	81.45	79.95
Value of n.....	1.19	1.18	1.03	1.02

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DETERMINATION OF TAR.

In the experiments described above all tar yields were determined by difference; consequently all errors were thrown on this part of the volatile matter. The yields of tars—that is, products other than gas and water—indicated by this method were very high. Some experiments were undertaken to determine tar yields directly, the determination of gas being neglected.

YIELDS OF TAR BELOW 500° C.

The apparatus that is shown in figure 9 was constructed of glass. The coal was contained in the large bulb *a*, the heavier tars were condensed in the bulb *b*, and most of the oils and water were obtained in the U tube *d*, which was cooled with cracked ice. Further condensate was collected in the weighed tube *e*, which was cooled with a freezing mixture at a temperature of $-10°$ to $-15°$ C. The P_2O_5 bulb retained any water escaping from the condensing system, and its contents were protected by a tube filled with calcium chloride.

Runs were made both at reduced and atmospheric pressure. The bulb *a* was first filled with the coal and then sealed on the apparatus. Before a run was started the air was pumped out and the apparatus filled with natural gas (saturated hydrocarbons). The coal was then heated slowly during 4 to 6 hours up to about 475° C., and in some cases was held at that temperature for several hours longer. Most of the tar collected in the bulb *b*. In tests made at reduced pressure (25 to 50 mm.) all of the water was kept out of this bulb by occasional warming with a flame. At the end of a run the glass connections to the bulb were cut off close to the retort and just under the manometer. The bulb and contents were weighed, the tar was dissolved out, and the bulb reweighed. In the runs at atmospheric pressure water could not be kept out of the bulb, but was separated from the tar at the end of the run by mixing with benzol and distilling off.

Any oil and water that collected in the U tube *d* were drained off into separate bottles by opening the drip cock *f* and weighed. The total increase in weight of the cooled tube *e* was determined, the contents mixed with benzol, and the volume of water read off in a burette. The total condensate obtained in *e* was very small. The increase in weight of the P_2O_5 tube was small, and was considered as water. The results are presented in Table 13 following.

The volatile matter in the residue was determined by heating about 5 grams in the inverted retort (see fig. 7) in a vacuum to above 1,000° C. A cotton plug was placed in the lower end of the quartz tube to filter out the tar, and the increase in weight of tube and cotton showed that only traces of tar were produced in most cases from the residues.

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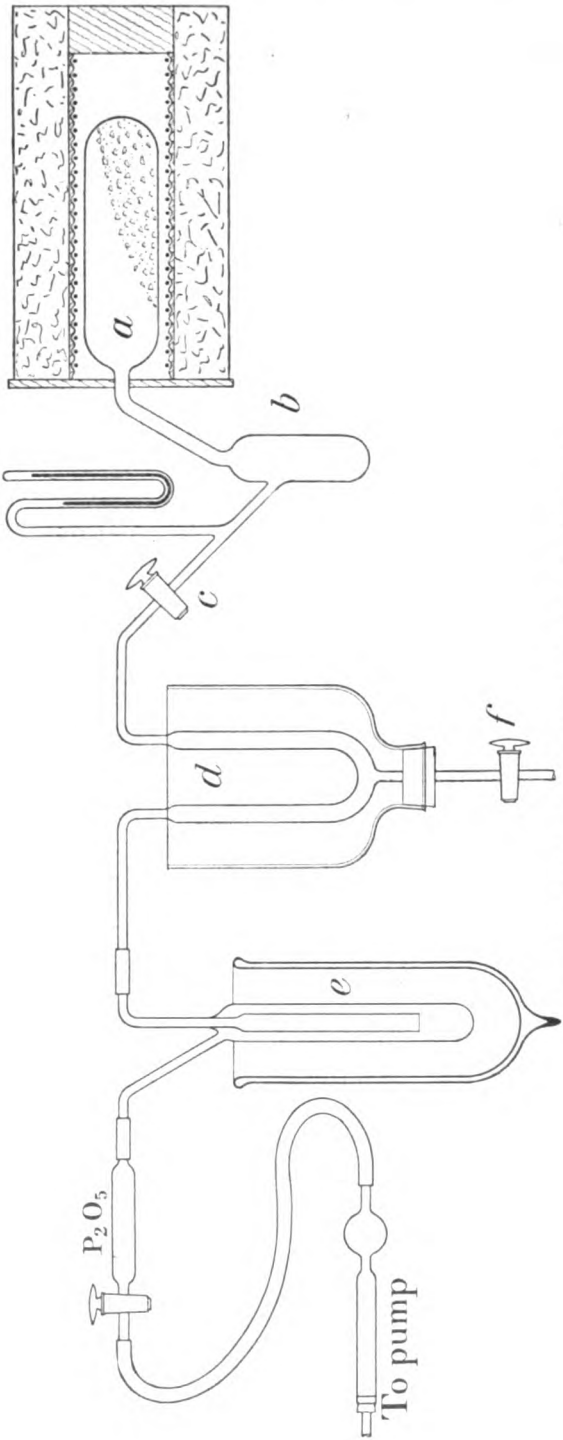

FIGURE 9.—Apparatus for distilling tar from coal at low temperatures.

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TABLE 13.—*Results of direct determinations of yields of tar at temperatures below 500° C.*

Experiment No.	Coal.	Pressure used.	Weight distilled.	Volatile matter.	Water.	Tar.	Residue.
			Grams.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
143	Pittsburgh (No. 650)	Vacuum	155.3	21.35	4.50	11.10	78.65
147	do	do	115.9	21.70	4.35	12.15	78.30
148	do	do	155.0	19.55	3.70	11.55	80.45
157	do	Atmospheric	104.1	17.00	4.05	8.65	83.00
150	Utah (No. 25) [a]	Vacuum	129.5	.....	10.65	10.25	.....
152	do	Atmospheric	134.6	27.80	10.20	9.75	72.20
153	Utah (No. 136) [b]	do	140.4	32.50	15.85	5.10	67.50
156	Illinois (No. 39) [c]	do	136.0	24.40	8.30	11.20	75.60
168	Illinois (No. 48)	Vacuum	104.0	23.85	12.20	7.70	76.15
166	do	Atmospheric	99.8	23.05	12.25	5.60	76.95
162	New River (No. 728) [d]	Vacuum	128.8	10.25	1.50	4.75	89.75
165	do	Atmospheric	115.3	7.90	1.75	3.00	92.10
161	Oklahoma (Lab. No. 19727) [e]	Vacuum	107.0	25.20	4.15	16.00	74.80
160	do	Atmospheric	91.5	22.20	5.20	12.00	77.80

Experiment No.	Solid residue. Character.	Content of volatile matter, per cent at 1,000° C. Total.	H_2O.	Tar.
143	Fused and slightly swelled	10.30	2.70	0.30
147	do	13.85	3.45	.55
148	do	.....	.....	.....
157	Fused and swelled	.....	.....	.....
150	Not coked	.....	.....	.....
152	do	14.15	2.90	Trace.
153	do	15.80	1.90	Trace.
156	Fused and swelled	15.50	4.85	Trace.
168	Not coked	.....	.....	.....
166	do	.....	.....	.....
162	Slightly coked	.....	.....	.....
165	do	8.30	2.15	Trace.
161	Fused, not swelled	14.60	4.70	Trace.
160	do	14.20	4.15	.25

[a] Castle Gate. [b] Coalville. [c] Harrisburg (Saline County), Ill. [d] Eccles, W. Va. [e] Pittsburg County, lower Hartshorne seam.

TAR YIELDS AT HIGH TEMPERATURES.

METHOD AND APPARATUS.

Tar yields at higher temperatures were obtained by distilling the coal in a current of gas to sweep out the tar vapors as fast as formed, and determined by direct weighing. The results shown in Table 14 were obtained with a current of dry nitrogen. A clay tube with a tapered outlet [a] was fixed in an upright position with the tapered end down. It was heated along its central zone for about 10 cm. in an electric furnace. The coal, 20 to 40 mesh in size, was placed in an iron-gauze capsule, which was hung on a stiff wire so arranged that the coal could be plunged suddenly into the heated part of the tube while the nitrogen was passing through. The tar liberated as characteristic tar mist was passed through U tubes filled with absorbent cotton and then over P_2O_5, the whole being heated to 105° to 110° C. This temperature prevented condensation of water in the cotton and caused

[a] Johnson, C. M., A vitrified clay combustion tube: Jour. Ind. and Eng. Chem., vol. 5, 1913, pp. 488, 581.

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the dehydrated lighter oils to pass through into another tube cooled to 0° C. with ice, where the oils were largely condensed. Any tar remaining in the clay tube was extracted with acetone.

Plate I, A, shows an apparatus in which 200-gram samples of Pittsburgh coal (No. 650) were distilled in a rapid current of the gas generated by the coal. The coal was placed in the iron-gauze basket, which was fastened to a ¼-inch pipe, in such a way that the closed end of the pipe was buried in the center of the coal. The pipe served as a thermoelement jacket, and slid through a rubber stopper in the top of the 2-inch still so that the coal could be lowered quickly into the heated zone. Before starting a run the air was displaced by Pittsburgh natural gas (saturated hydrocarbons). The gases were circulated from the top downward by means of a rotary pump (Gaede box vacuum pump), the excess gases of distillation escaping through the side valve. Tar and water were caught in the three filtering flasks and in the cotton tube. The flask under the still contained water to cool the gases. The cotton, after the run, was placed in a flask and the water distilled off through a condenser. The $CaCl_2$ tower protected the pump from water. It was not an easy matter to separate tar and water, but a fairly satisfactory separation was obtained and the results, shown in Table 14, are fairly accurate.

RESULTS OF TESTS.

The table shows that rapid heating tends to produce more tar and less coke than slow heating. The coke from rapid heating was dense and the coal had shrunk (*1*, Pl. I, B), while that from the slow heating was very porous. The coal had fused, swelled out the lower end of the gauze basket and then shrunk (*2*, Pl. I, B.).

TABLE 14.—*Results of heating Pittsburgh coal (No. 650) to high temperatures, showing variation in yield of tar and volatile matter with different rates of heating.*

DISTILLATION IN CURRENT OF NITROGEN.

Experiment.	Weight of coal.	Volatile matter.	Tar.	Rate of heating.	Total time.	Maximum temperature.
No.	*Grams.*	*Per ct.*	*Per ct.*		*Hr. min.*	*° C.*
129	6.954	28.8	9.7	Heated to 750° C. in 10 minutes, to 1,000° C. in 16 minutes	30	1,030
130	6.836	32.3	11.4	Heated to 710° C. in 10 minutes, to 1,000° C. in 20 minutes	40	1,050
133	8.400	26.5	9.3	Heated to 700° C. in 200 minutes, to 1,000° C. in 300 minutes	5 15	1,040
131	7.519	30.3	11.7	Plunged into hot tube at 1,010° C	20	1,050
132	8.718	30.4	10.5	Plunged into hot tube at 1,030° C	25	1,040
DISTILLATION IN CURRENT OF COAL GAS.						
137	209.0	32.00	9.95	Heated to 500° C. in 45 minutes, to 950° C. in 1½ hours	5	950
139	235.7	31.50	10.45	do	5	1,010
136	223.0	34.75	12.05	Plunged into hot tube; heated to 1,000° C. in 15 minutes	30	1,020
138	189.9	36.20	11.90	Plunged into hot tube; heated to 960° C. in 15 minutes	30	980

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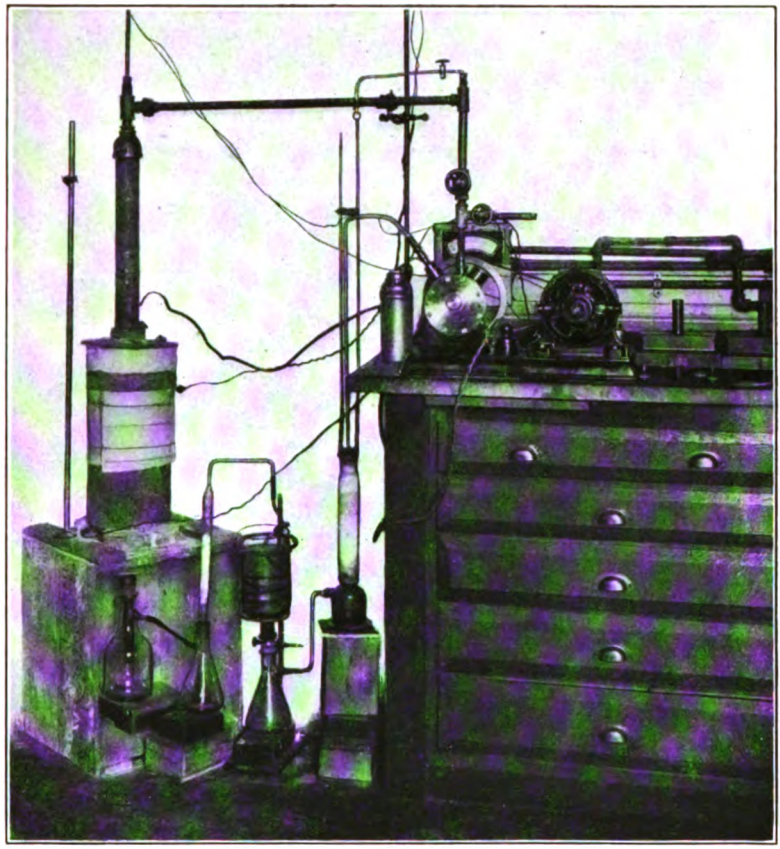
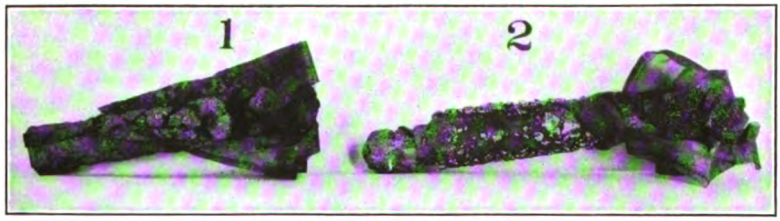


A. APPARATUS FOR CARBONIZING COAL IN A CURRENT OF ITS OWN GAS.



B. COKE FROM PITTSBURGH COAL CARBONIZED AT HIGH TEMPERATURES. 1, COKE FROM RAPID HEATING; 2, COKE FROM SLOW HEATING.

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RATE OF EVOLUTION OF VOLATILE MATTER.

Experiments were undertaken to determine how quickly the total volatile matter may be driven off from coal. This is an important question in firing boiler furnaces or others where coal is thrown into a fire box that is at a high temperature.

For determining the rate of evolution of total volatile matter, Pittsburgh coal (No. 650) and the apparatus shown in figure 10 were used. The closed end of a silica tube 6 mm. in internal diameter was inserted in an electric heater, in a horizontal position. Then 0.5 gram of coal was weighed carefully into a short section of glass tubing which was loosely attached to the silica tube by a piece of rubber tubing. The electric heater was swung on the pivots on which it had been mounted so that it could be quickly turned upright, causing the coal to fall into the heated end of the tube. After the required number of seconds the silica tube was withdrawn from the heater and plunged into cold water. Pittsburgh coal under these conditions fused so rapidly that there was no mechanical loss of coal particles. The residual coke was removed from the tube with a stiff wire and weighed on a watch glass. Care was taken to prevent the coke from becoming contaminated with tar condensed on the walls above the heated part.

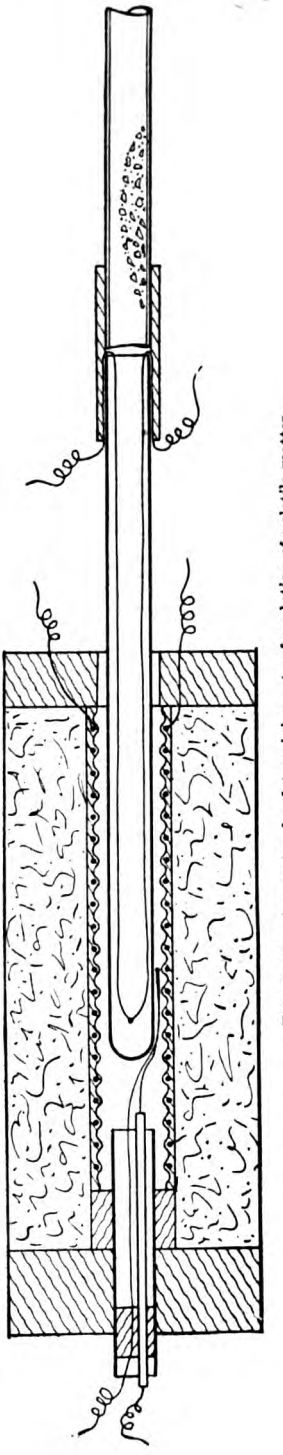

FIGURE 10.—Apparatus for determining rate of evolution of volatile matter.

RESULTS OF TESTS.

In the results shown in part A of Table 15 and in figure 11 only the temperature of the outside wall was taken; in the results shown in parts B and C of Table 15 and in figure 12 the temperature was taken within the coal by means of a thermocouple, made of fine wires arranged as shown in figure 10. The results shown in part B of the

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table and by the curve *a* in figure 12 were obtained by plunging the cold tube containing the coal into the hot furnace, in order to obtain a slower rate of heating. Slower rates than this were attempted but slow heating caused the coal to swell out of the hot tube.

The curves show that when a particle of coal has reached a temperature between 850° and 900° C. its carbonization is complete. The volatile matter at temperatures above 900° C. is driven off within a fraction of a second after the coal reaches that temperature. The rate of distillation, then, on the grate of a boiler furnace or elsewhere is determined by the rate at which heat is supplied to the green coal and its temperature raised. Coal is a poor conductor of heat and becomes heated very slowly when in thick layers. Hence, when coal is subjected to a high temperature, as in a boiler furnace, the thinner the layer the more rapidly volatilization takes place.

TABLE 15.—*Results of experiments showing rate of evolution of volatile matter.*

A. COAL DROPPED IN HEATED TUBE, TEMPERATURE 900° TO 1,000° C.

Temperature.	Volatile matter evolved in—											For curve see—
	1 second.	5 seconds.	10 seconds.	15 seconds.	20 seconds.	30 seconds.	45 seconds.	60 seconds.	90 seconds.	120 seconds.	180 seconds.	
1,000 [a]	7.20	13.80	22.60	27.55	28.25	31.30	31.40	32.50	.....	[b] 33.10		Fig. 11
900 [a]	.....	.....	.....	22.35	27.30	28.10	30.00	[b] 30.40				Fig. 11

B. COLD TUBE PLUNGED IN FURNACE HEATED TO 1,080° C.[c]

1,080	.....	.....	.....	.....	14.10	.....	27.50	32.45	34.20	.....	.....	34.40	

C. COAL DROPPED IN HEATED TUBE, TEMPERATURE 860° TO 1,120° C.[c]

860	.....	10.80	.....	23.00	.....	29.80	[d] 30.85	31.45	.....	.....	31.70	*b*, Fig. 12
970	6.45	16.90	22.90	.....	30.10	.....	[d] 33.10	.....	.....	.....	32.55	*c*, Fig. 12
1,080	.....	20.20	30.60	.....	33.35	33.75	.....	.....	.....	.....	34.30	*d*, Fig. 12
1,120	12.90	25.65	31.90	33.52	.....	34.80	.....	34.15	.....	.....	34.05	*e*, Fig. 12

a Temperature measured on wall of tube.
b End of test.
c Temperature measured in the coal.
d 40 seconds.

DISCUSSION AND GRAPHIC PRESENTATION OF RESULTS.

Figures 13 to 16 show graphically the results of experiments in which samples of the four coals tested were heated at temperatures less than 500° C. The curves show the distribution of the volatile matter as tar, water, and gases in the coals. The Eastern coals yielded proportionately more tar and less water than the younger Western coals.

The total yield of gas from each of the four coals, in cubic centimeters per 100 grams of coal, is shown in figure 17. The yields of

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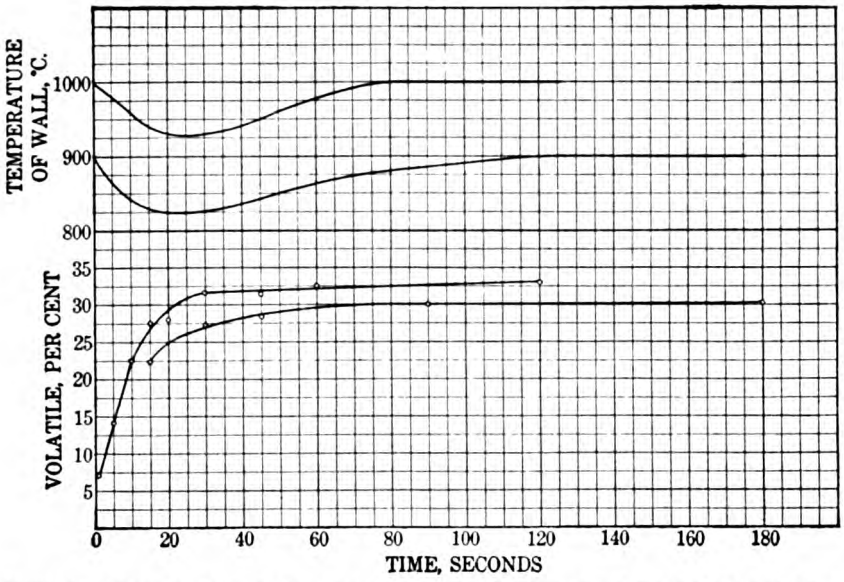

FIGURE 11.—Curves showing rate of evolution of volatile matter from coal placed in highly heated tube, also temperature of wall.

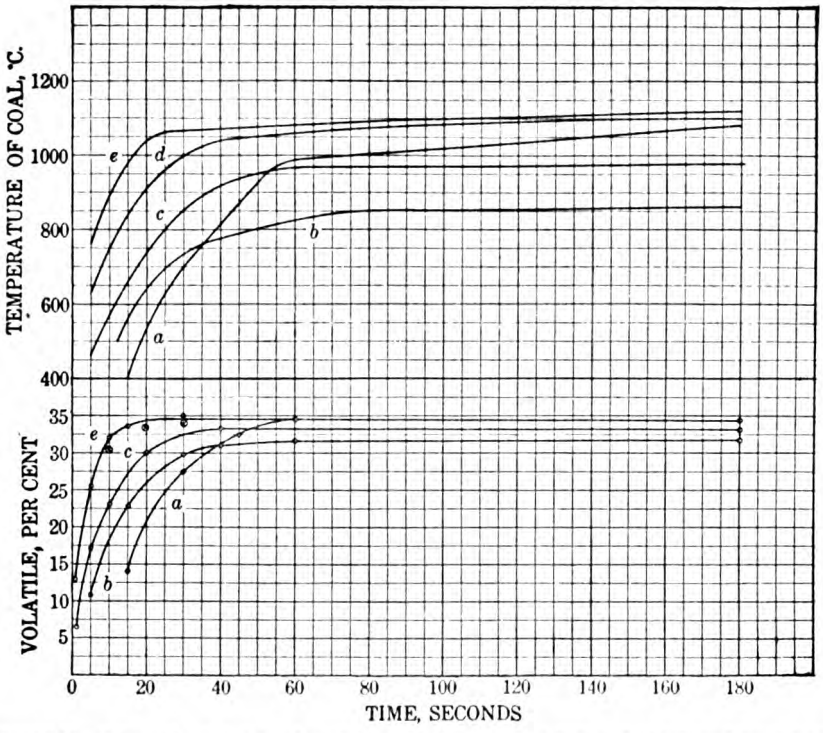

FIGURE 12.—Curves showing rate of evolution of volatile matter from coal at various temperatures, also temperature of coal.

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carbon dioxide, carbon monoxide, unsaturated hydrocarbons, saturated hydrocarbons, and hydrogen in liters per kilogram of coal, are shown in figures 18 to 21. The yields are represented by straight

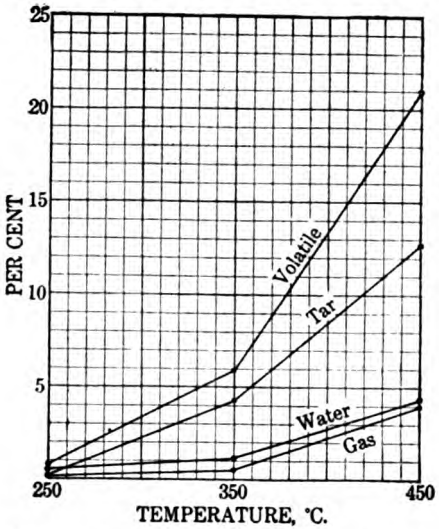

FIGURE 13.—Curves showing proportions of tar, water, and gas in volatile matter distilled from Pittsburgh coal at temperatures of 250° to 450° C.

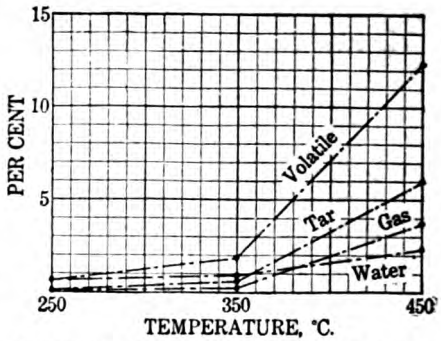

FIGURE 14.—Curves showing proportions of tar, water, and gas in volatile matter distilled from New River (W. Va.) coal at temperatures of 250° to 450° C.

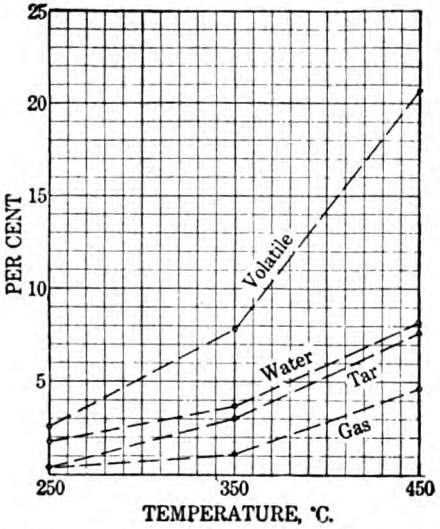

FIGURE 15.—Curves showing proportions of tar, water, and gas in volatile matter distilled from Illinois coal at temperatures of 250° to 450° C.

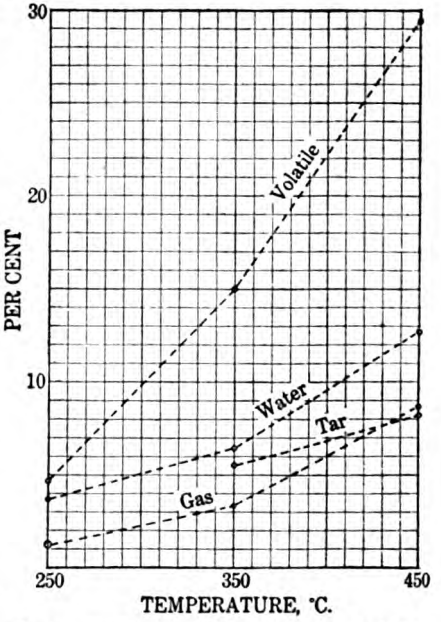

FIGURE 16.—Curves showing proportions of tar, water, and gas in volatile matter distilled from Wyoming coal at temperatures of 250° to 450° C.

lines. The large percentage of saturated hydrocarbons in the gas from the older Appalachian coals should be noted. The younger Western coals, on the other hand, decompose more easily and yield

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at first large amounts of CO_2. The older Eastern coals also yield large quantities of CO_2 in the first fractions. These results were all obtained in less than 15 hours' heating.

The volumes of gases distilled from Pittsburgh (No. 650) and Wyoming (No. 43) coal when heated for longer periods at 350° C. are shown in figures 22 and 23. Figure 24 shows that much more volatile matter was obtained by further heating at 450° C. At the end of long periods of heating at this temperature hydrogen becomes the predominating constituent of the gas, and continues to be on heating to higher temperatures (see p. 25).

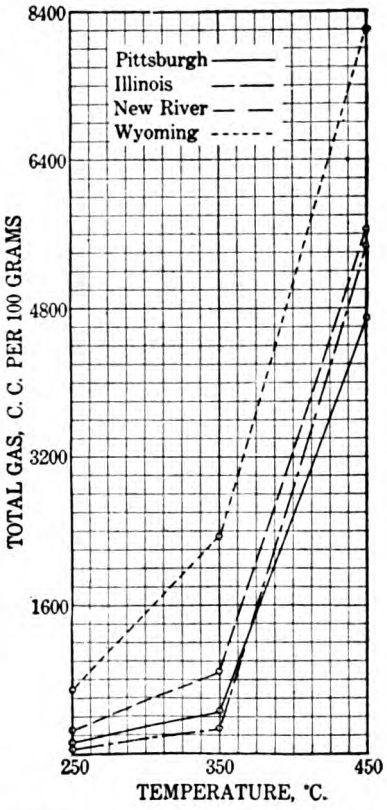

FIGURE 17.—Curves showing relative volumes of gas from four coals.

SECONDARY REACTIONS FROM SUPERHEATING OF VOLATILE MATTER.

The general effect of secondary reactions caused among the gases and tar vapors by superheating may be seen from Tables 11 and 12. At temperatures between 700° and 800° C. even brief contact with a hot surface caused considerable alteration. This is graphically shown in figure 25, in which the lengths of the straight lines represent yields of gases in cubic centimeters per 100 grams as obtained by a normal low-temperature distillation at 475° C. and from similar tests in which the volatile matter was heated to higher temperatures. The saturated hydrocarbons were calculated to methane (CH_4) and ethane (C_2H_6) in order to show the effect of heat in breaking down higher members in the series to lower. The chart was plotted from the results of runs 120 to 126 (Table 12), in which cracking of the tar vapors occurred at low pressure.

It will be noted that at 800° C. the yield of saturated and unsaturated hydrocarbons and the yield of hydrogen were largely increased, indicating cracking of hydrocarbon vapors. In this connection it may be noted that Whitaker and Rittman[a] have shown

[a] Whitaker, M. C., and Rittman, W. F., Thermal reactions in carbureting water gas: Jour. Ind. and Eng. Chem., vol. 6, 1914, pp. 383-392, 472-478.

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theoretically and experimentally that the yield of hydrocarbon gases obtained by "cracking" oil vapors at a given temperature is increased by reducing the pressure, whereas the yield of hydrogen is

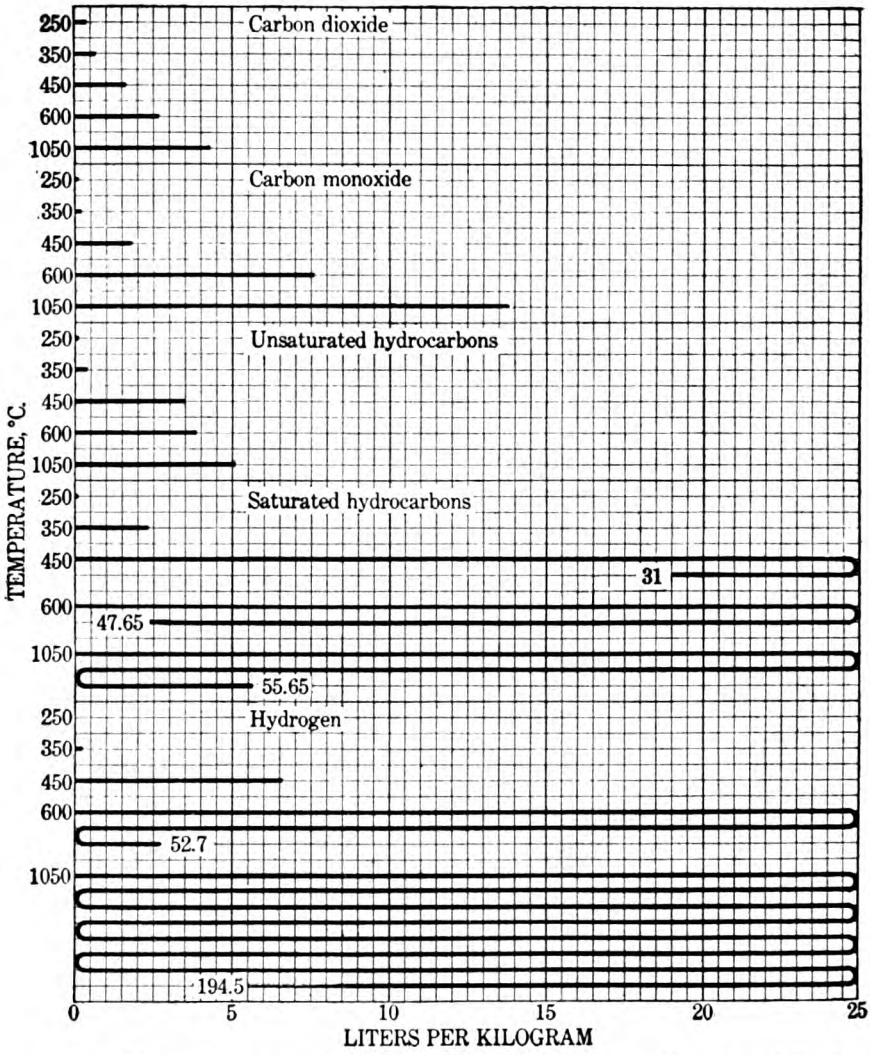

FIGURE 18.—Volumes of gaseous constituents in volatile matter distilled from Pittsburgh coal (No. 650) at temperatures of 250° to 1,050° C.

decreased. In the experiments described in this paper increasing the surface of contact in the superheating section of the tube doubled the hydrogen yield at 800° C., but had little effect on the yields of other gases. It will be noted that the superheating section was short

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and the time of contact correspondingly brief. It seems likely that at temperatures above 750° C. reactions can occur so rapidly that obtaining the volatile matter from coal in the form in which it is

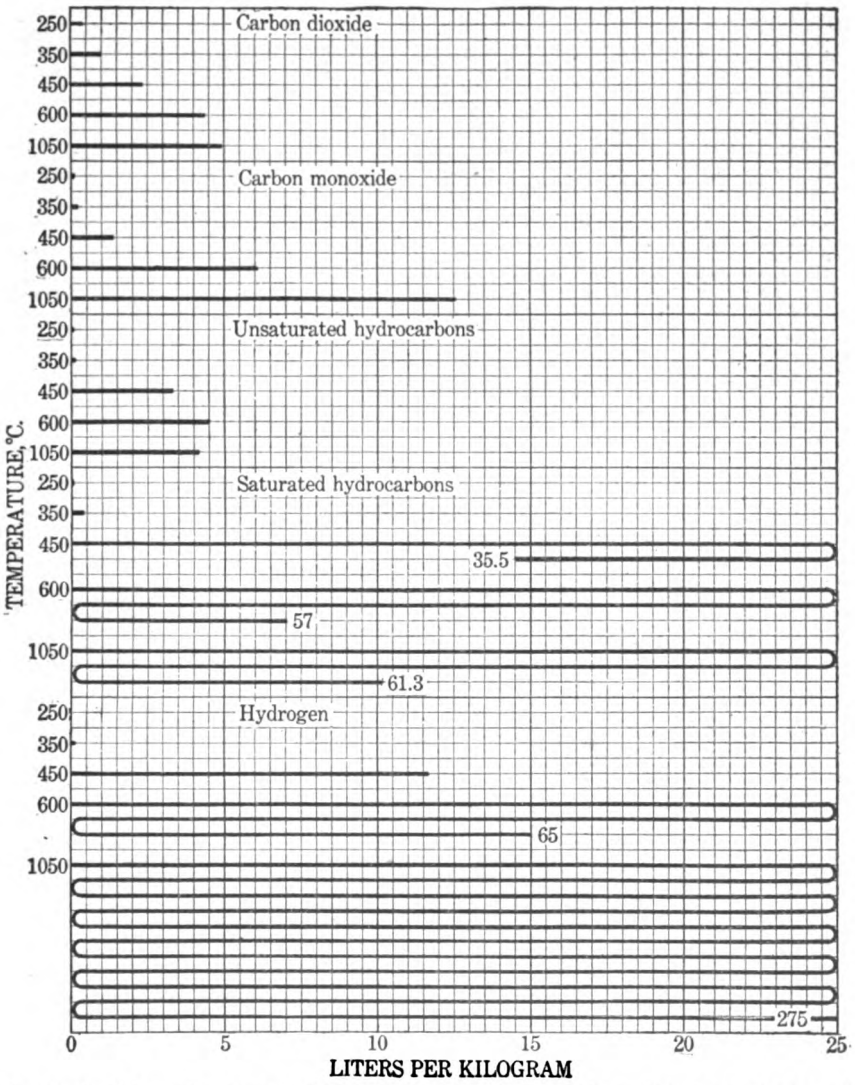

FIGURE 19.—Volume of gaseous constituents in volatile matter distilled from New River, W. Va., coal (No. 674) at temperatures of 250° to 1,050° C.

liberated is impossible at such high temperatures. This important fact should be kept in mind in attempting any interpretation of data on distilling coal at high temperatures.

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SUMMARY OF RESULTS OF EXPERIMENTS.

The data obtained on the distillation of volatile matter from coal in the experiments described in this report may be summarized as follows:

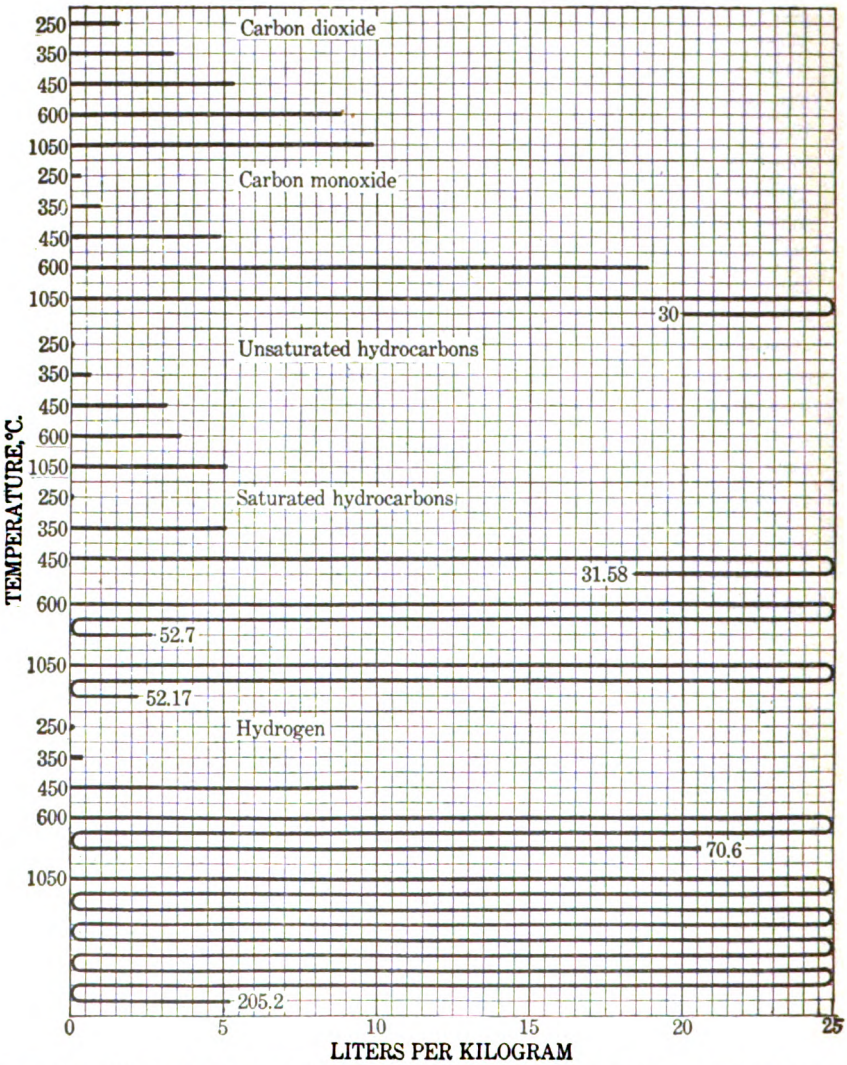

FIGURE 20.—Volume of gaseous constituents in volatile matter distilled from Illinois coal (No. 48) at temperatures of 250° to 1,050° C.

VOLATILE MATTER.

1. More than two-thirds of the organic substance of coal is decomposed at temperatures below 500° C. (932° F.). Different kinds of coal vary in ease of decomposition, the older coals of the Appa-

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lachian field being less easily and rapidly broken down than the younger coals of the Middle-Western and Western fields.

2. The first decomposition occurring in any type of coal as the temperature is raised is the breaking down of certain oxygen-con-

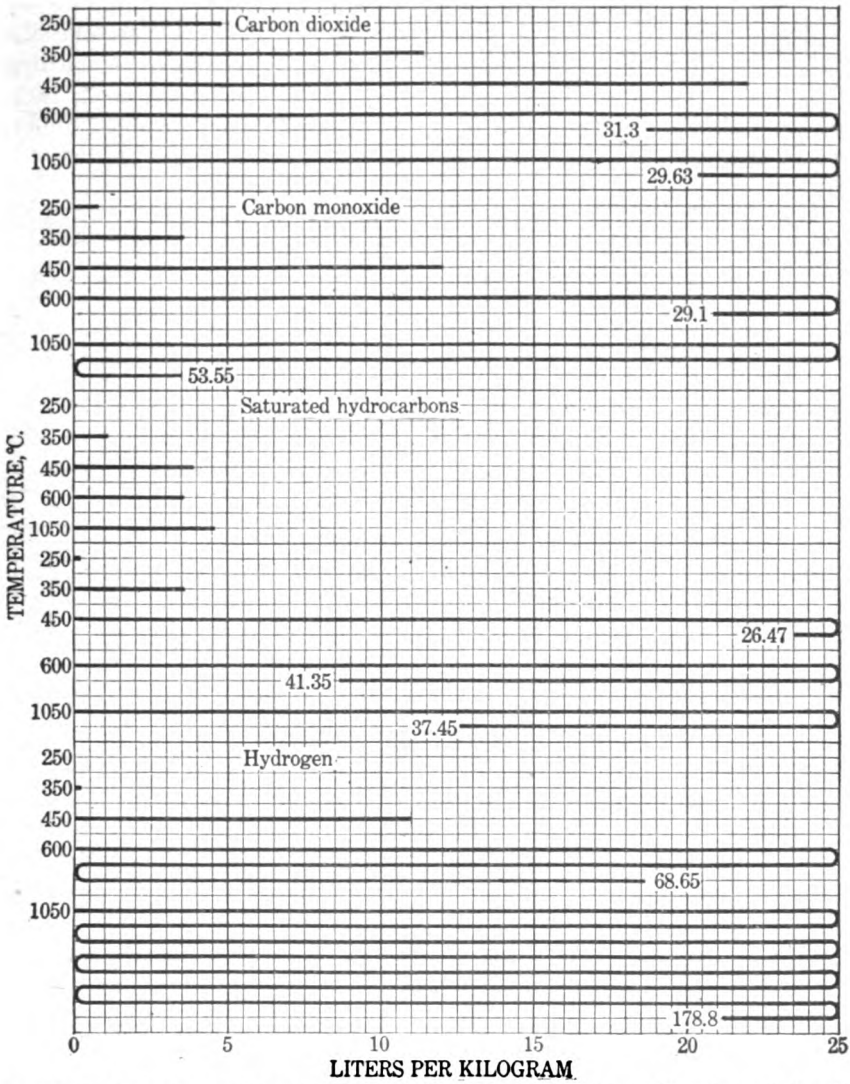

FIGURE 21.—Volume of gaseous constituents in volatile matter distilled from Wyoming coal (No. 43) at temperatures of 250° to 1,050°C.

taining substances related to cellulose, the products being chiefly water, CO_2, and CO.

3. Coal probably breaks down more or less at all temperatures, but the temperature at which reactions occur to any extent in experimental time is considerably above normal atmospheric temperatures.

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With bituminous coal of the Pittsburgh type volatile matter begins to distill in appreciable quantities at 250° C. (482° F.), and with Western coals, especially the subbituminous types, at a still lower temperature.

4. The formation of liquid and gaseous paraffin hydrocarbons begins at an early stage, but proceeds slowly at the lower temperatures. At 450° C. (842° F.) the volatile matter from the maturer bituminous coals consists chiefly of paraffins, whereas coals more nearly subbituminous, such as the Wyoming type, produce chiefly water and carbon dioxide at temperatures up to at least 450° C.

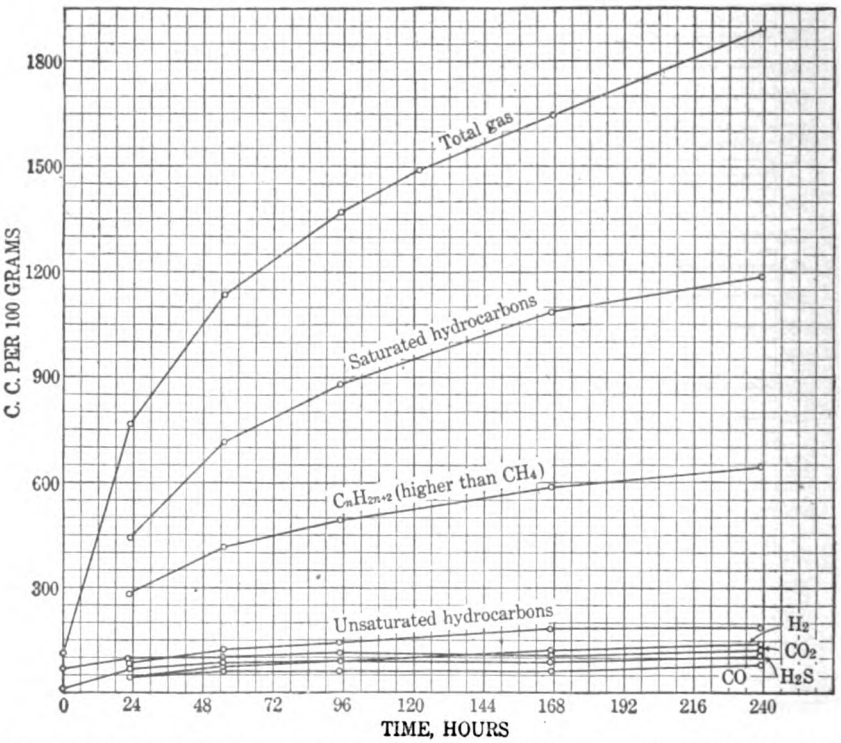

FIGURE 22.—Curves showing volumes of gases distilled from Pittsburgh coal (No. 650) heated for long periods at 350° C.

5. Paraffin hydrocarbon gases of the series C_nH_{2n+2} higher than CH_4 predominate at temperatures below 400° C.

6. Thermal decomposition of the volatile matter takes place very easily at temperatures above 750° C. (1382° F.) and increases the yield of the permanent gases, hydrogen and hydrocarbons.

7. Distillation at temperatures above 750° C. yields gases in which hydrogen predominates, whether secondary reactions are prevented or not, but secondary reactions of the volatile products will increase the total gas yield at the expense of the tar.

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8. The yields of tar from all those coals tested may be increased by distilling at lower temperatures than the usual practice of carbonization in bulk. For example, coal of the Pittsburgh type may be made to yield 13 per cent of its weight as tar at temperatures below 500° C., more than twice the amount obtained in commercial processes for carbonizing coal at high temperatures. Low-temperature tars are different in character from high-temperature tars.

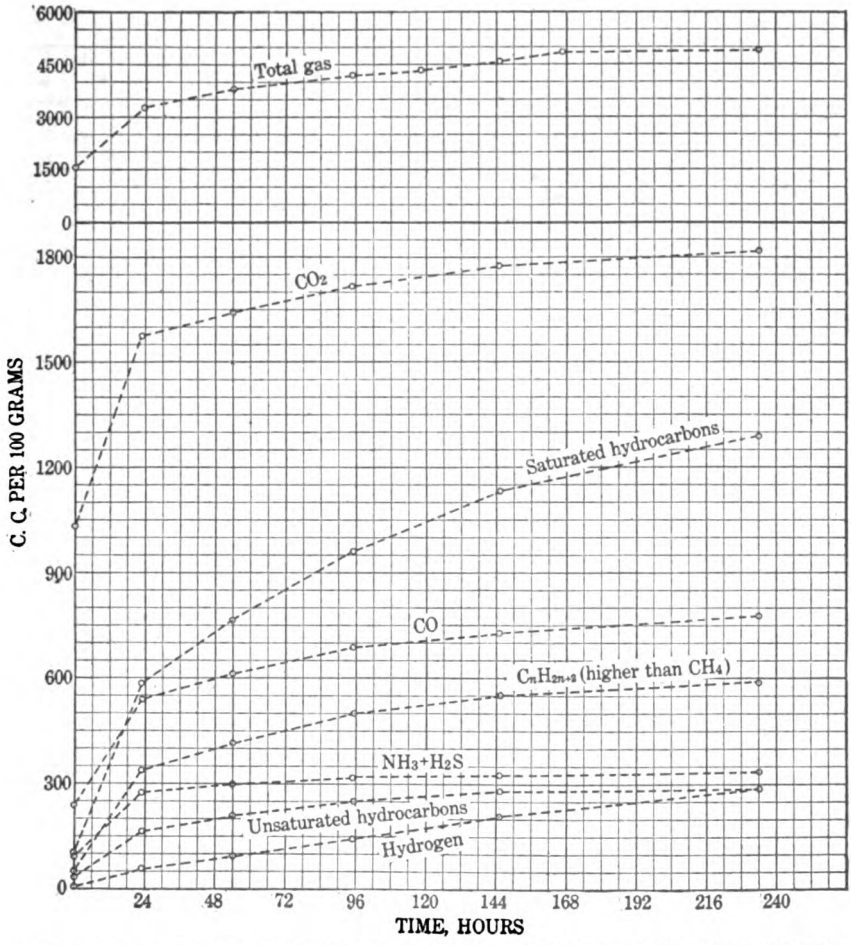

FIGURE 23.—Curves showing volumes of gases distilled from Wyoming coal (No. 43) heated for long periods at 350° C.

9. Water of decomposition is produced from coal at temperatures above about 250° C., but in greater amount below 500° C than above. This water vapor may react with tar vapors or gases in passing out of a heated retort during high-temperature carbonization.

10. Sulphurous gases (H_2S, etc.) are readily produced in the decomposition of coal; they begin to be formed at temperatures as

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low as 250° C. (482° F.), and as the temperature rises their production reaches its maximum more rapidly than does that of the hydrocarbons and hydrogen.

11. Bituminous coals of approximately equal volatile matter content but of different oxygen content, such as Pittsburgh coal (No. 650) and Illinois coal (No. 48) yield volatile products of very different character. The coal of higher oxygen content gives off at 450° C. a greater amount of water of decomposition, a less amount of tar, and a

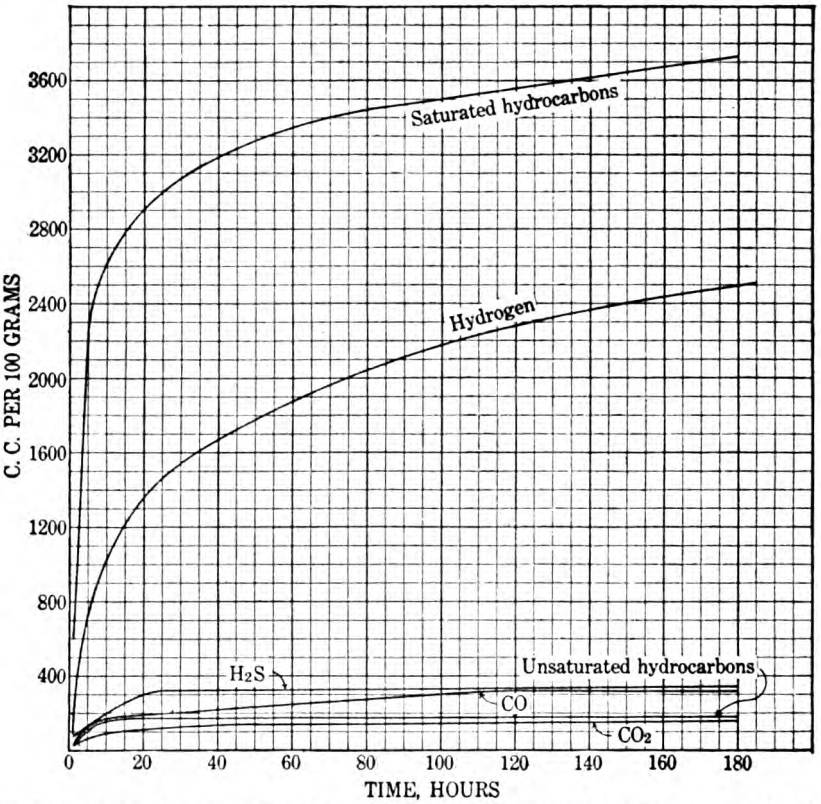

FIGURE 24.—Curves showing volumes of gases distilled from 350° C. residue of Pittsburgh coal (No. 650) heated for long periods at 450° C.

somewhat greater volume of gas, the excess gas consisting largely of CO_2 and CO. As a rule the higher the oxygen content in the coal the less will be the proportion of hydrocarbons and tar in the volatile matter.

12. The volatile matter is practically all driven off from Pittsburgh bituminous coal in the time required to heat it to 900° C. This time may easily be as short as five seconds under favorable conditions, but in practice depends upon the thickness of the coal layer and the temperature gradient.

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SOLID RESIDUE OR COKE.

13. Small-scale experiments are generally unreliable tests for quality of coke. However, the following facts may be noted:

Slow heating of Pittsburgh bituminous coal (a good coking coal) at atmospheric pressure causes swelling of the coal at the fusion temperature and a light, porous, inferior grade of coke, but slow heating in a vacuum (pressure less than 30 mm.) produces a dense coke.

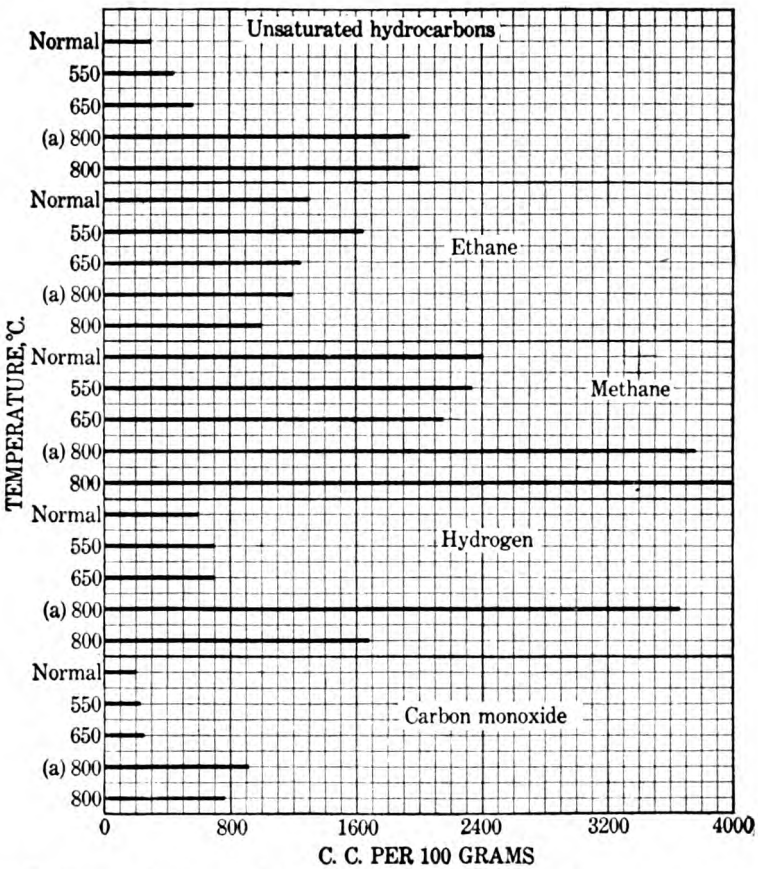

FIGURE 25.—Diagram showing secondary reactions among volatile products caused by heating at temperatures above 475° C.

Heating the coal rapidly to 900° C. or higher at a pressure 1 atmosphere or less also produces dense, hard coke of superior quality. Heating this coal at 350° C. for a long time destroys its coking and fusing qualities entirely; the coal particles retain their sharp edges when heated to coking temperatures and look like the original coal but have a brighter luster.

Slow carbonization tends to increase the yield of coke.

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RECENT WORK OF OTHER INVESTIGATORS.

WORK OF WHEELER AND ASSOCIATES.

Wheeler and his associates, in England, have carried on an extensive series of destructive distillations of coal chiefly with a view to its bearing on the process of ignition or inflammation of coal dust suspended in air. In the first experiments [a] coal was distilled from a small platinum retort at fixed temperatures ranging from 450° to 1100° C., the gases being collected at atmospheric pressure. Yields of tar, gas, and residue were determined. From these experiments it was concluded that coal has a well-defined decomposition point between 700° and 800° C. which corresponds with a marked increase in the quantity of hydrogen evolved. In a continuation of the same work,[b] the authors distilled coal from the same apparatus as before, the gas being collected in 5-second intervals, to obtain data on the rate of evolution and the composition of the several fractions. Coal was also distilled in a vacuum, fractions being collected at 50° intervals up to a temperature of 650° C.

The conclusion drawn was that coal probably consisted of two main types of compounds—one more unstable and yielding mostly paraffin hydrocarbons; the other decomposing with greater difficulty and yielding mostly hydrogen. In further support of this view, Clark and Wheeler [c] separated coal into two parts—a pyridine extract and an insoluble residue. Distillations were made on the original coal, extract, and residue. By the use of chloroform as an additional solvent, the coal was separated into "humus" and "resinous" substances. The "humus" bodies were regarded as degradation products of cellulose and as being responsible for hydrogen in the gaseous volatile matter.[d]

In general, the conclusions arrived at by the authors as a result of the experimental work described in this report do not accord with the views of Wheeler as to the relative ease of decomposition of the two classes of constituents in coal.

Distillations of a larger quantity of coal (1 to 2 kilos) in a vacuum were also made in Wheeler's laboratories, for gases [e] and liquid products,[f] at temperatures below 500° C. The results on gases sub-

[a] Burgess, M. J., and Wheeler, R. V., The volatile constituents of coal: Jour. Chem. Soc., vol. 97, 1910, pp. 1917-1935.

[b] Burgess, M. J., and Wheeler, R. V., The volatile constituents of coal: Jour. Chem. Soc., vol. 99, 1911, pp. 650-667.

[c] Clark, A. H., and Wheeler, R. V., The volatile constituents of coal: Jour. Chem. Soc., vol. 103, 1913, pp. 1705-1715.

[d] For discussion, see Porter, H. C., and Taylor, G. B., The mode of decomposition of coal by heat: Proc. Am. Gas Inst., vol. 9, 1914 p. 238.

[e] Burgess, M. J., and Wheeler, R. V., The distillation of coal in a vacuum: Jour. Chem. Soc., vol. 105, 1914, pp. 131-140.

[f] Jones, D. T., and Wheeler, R. V., The composition of coal: Jour. Chem. Soc., vol. 105, 1914, pp. 140-151, 2562-2565.

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stantially accord with the results of the experiments described in this report. The low-temperature tar consisted of about 50 per cent pitch and 50 per cent of oil, distilled from the tar at temperatures below 300° C. The composition of the oil was as follows: Ethylenic hydrocarbons, 40 to 45 per cent; phenols (chiefly cresol and xylenols), 12 to 15 per cent; aromatic compounds, 7 per cent; a small amount of solid paraffin hydrocarbons; traces of pyridine bases; no benzene, anthracene, carbon disulphide, or solid aromatic hydrocarbons.

PICTET AND BOUVIER.

Pictet and Bouvier [a] obtained by distilling coal in a partial vacuum at 450° C. a thin tar containing apparently no phenols, naphthalene, anthracene, or other aromatic compound.

VIGNON.

Vignon [b] fractionally distilled several types of coal and found that paraffin gases predominated below 600° and hydrogen above 800° C.

RAU AND LAMBRIS.

Rau and Lambris [c] have conducted a thorough investigation of the formation of water in the destructive distillation of fuels. They found that more water was produced by slow heating than by rapid heating. Fuels of greater maturity produced less water than younger, less altered fuels.

PARR AND OLIN.

Parr and Olin [d] treated Illinois coal with superheated steam, temperature 400° to 450° C., and obtained a yield of tar nearly equal to that of high-temperature carbonization; the gas yield was less than 10 per cent. Seventy per cent of the tar boiled below 240° C. and contained little free carbon. In a more recent report of the same authors [e] a good semicoke was described, which was made at 400° to 500° C. by a similar process. The tar produced contained only 30 per cent pitch with little free carbon. The oils were high in tar acids, but contained no naphthalene or anthracene.

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[a] Pictet, A., and Bouvier, M., Sur la distillation de la houille sous pression rèduite (Distillation of coal under reduced pressure): Compt. rend., t. 137, 1913, pp. 779–781; Gas World, vol. 60, 1914, p. 45; Ber. Deut. chem. Gesell, Jahrg. 46, 1913, pp. 3342–3353.

[b] Vignon, L. M., [Fractional distillation of coal]: Compt. rend., t. 155, 1912, pp. 1514–17; Jour. Gas Lighting, vol. 121, 1913, p. 107; Jour. Soc. Chem. Ind., vol. 32, 1913, p. 76.

[c] Rau, O., and Lambris, G., Formation of water in the dry distillation of fuels: Gas World, vol. 59, 1913, p. 239; Jour. Gasbel., Jahrg. 56, 1913, pp. 533, 557, 589.

[d] Parr, S. W., and Olin, H. L., Coking of coals at low temperaturess: Univ. Illinois Eng. Exp. Sta. Bull. 60, 1912, p. 12; Jour. Gas Lighting, vol. 122, 1913, pp. 533–535, 595–598.

[e] Parr, S. W., and Olin, H. L., The coking of coal at low temperature with special reference to the properties and compositions of the products: Univ. Illinois Eng. Exp. Sta. Bull. 79, 1915, pp. 29–30.

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LEWES.

Lewes [a] gives the composition of the tar from a low-temperature industrial operation. It contained an abundance of light solvent oils, small percentages of aromatic compounds and phenol, a high percentage of cresols, etc., no naphthalene, a small percentage of anthracene, and less than 2 per cent of free carbon.

HOLLINGS AND COBB.

The thermal effects of carbonization have recently been investigated by Hollings and Cobb.[b] They found that cellulose showed a strong exothermic reaction at temperatures of 345° to 400° C.; lignite showed the same effect, but it was less marked; and older coals, which have lost much of their cellulosic constituents, showed it scarcely at all. At temperatures betwen 600° and 800° C. all the types of coal tested displayed exothermicity; above 800° C. the process was neutral or slightly endothermic. Thermal effects at lower temperatures were only marked in coals of high oxygen content.

GENERAL CONCLUSIONS.

THE PRIMARY DECOMPOSITION PROCESSES.

A high-grade bituminous coal of the gas-making type decomposes by heat primarily into paraffin hydrocarbons and a completely altered nonvolatile residue, with small quantities of water, CO_2, and CO. The three latter products are the first produced, although in small quantity; from some other types of bituminous coal they are produced in greater relative quantities than from the gas-coal type. Complex and varied secondary reactions induced by superheating the hydrocarbons, water vapor, and CO_2 are of great importance in industrial high-temperature carbonization.

The products of low-temperature carbonization from coal of the Pittsburgh type on an industrial scale at about 800° to 900° F. (427° to 482° C.) will consist of a rich gas amounting to 0.6 to 0.7 cubic foot per pound of coal, and a large yield of oil, or tar, comprising 10 to 12 per cent of the coal. This tar consists chiefly of paraffin hydrocarbons, is very low or possibly entirely devoid of benzene and naphthalene derivatives and practically devoid of free carbon. The gas will contain 6 to 7 per cent of unsaturated hydrocarbons and 20 to 25 per cent of ethane and its higher homologues, and consequently will have a high calorific and illuminating value. The tar may be either redistilled or subjected to cracking processes so as

[a] Lewes, V. B., Carbonization of coal: Iron and Coal Trades Rev., vol. 83, 1911, p. 1011.

[b] Hollings, H., and Cobb, J. W., Thermal study of the carbonization process: Jour. Chem. Soc., vol. 107, 1915, p. 1106.

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to produce light oils—gasoline substitutes—whose yield will be greater than, and probably at least double, that obtained by high-temperature carbonization.

The results suggest the possibility that low-temperature carbonization might be utilized in gas manufacture as an enriching process by passing through the low-temperature retorts a relatively large quantity of a thin gas such as "blue-water gas," sweeping out the light oil vapors that are primarily liberated at these low temperatures. The utilization of inferior coals for making gas by such a process may be found practicable.

THE CONSTITUTION OF COAL.

Based on the experimental results of low-temperature decomposition in a vacuum, the following hypothesis is proposed for the constitution of coal:

All kinds of coal consist of cellulosic degradation products more or less altered by the processes of aging, together with derivatives of resinous substances in different proportions, also more or less altered. These substances are many in number and closely graded into one another in their nature and composition. They all undergo decomposition on moderate heating; some, however, decompose more rapidly than others at the lower temperatures. The less altered cellulosic derivatives decompose more easily than the more altered derivatives and also more easily than the resinous derivatives. The cellulosic derivatives on moderate heating decompose so as to yield water, carbon dioxide, carbon monoxide, and hydrocarbons, giving less of the first three products the more mature and altered they are. The resinous derivatives, on the other hand, decompose on moderate heating so as to yield principally the paraffin hydrocarbons, with probably hydrogen also as a direct decomposition product.

The more mature bituminous coals with good coking properties contain a large percentage of resinous derivatives, and their cellulosic constituents have been highly altered. The younger bituminous and subbituminous coals consist chiefly of cellulosic derivatives much less altered than those in the older coals. They undergo a large amount of decomposition below their fusion point and, partly for that reason, many of them do not coke.

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